

# ANALYTICAL ABSTRACTS

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## ANALYTICAL ABSTRACTS

## 1.—GENERAL ANALYTICAL CHEMISTRY

2034. Measurement of the effect of dilution on pH. R. G. Bates (*Anal. Chem.*, 1954, **26** [5], 871-874).—It is suggested that the effect of dilution or concn. of buffer on the pH of a soln. should be expressed numerically in terms of a unit, dilution value =  $\Delta\text{pH}_1$ , defined as the increase in pH caused by diluting the soln. with an equal vol. of dist.  $\text{H}_2\text{O}$ . The dilution value and the van Slyke buffer value together give a complete measure of the effectiveness of a soln. for controlling pH. Factors conducive to change of pH on dilution are discussed, and equations for the dilution values of strong acids, strong bases and buffer soln. are developed. Observed and calculated values of  $\Delta\text{pH}_1$  for HCl and a number of buffer solutions are listed.

W. J. BAKER

2035. Methyl purple vs. methyl orange in alkalinity titrations. R. W. Frey (*Water & Sewage Wks.*, 1954, **101** [5, Pt. 2], 140).—Methyl purple can be used to advantage in place of methyl orange in the determination of the alkalinity of sewage and non-potable waters. The end-point is sharp, and results are in satisfactory agreement with those obtained by the use of methyl orange. A. J. MEE

2036. Metal complexes in analytical chemistry. N. A. Gibson (*Rev. Pure Appl. Chem.*, 1954, **4** [1], 101-110).—This review article describes the uses of metal complexes as redox indicators or as masking reagents in the detection and isolation of metals. General requirements for these reagents in the detection and the determination of metals are discussed. The production of anionic complexes that, when combined with quaternary arsonium radicals of high molecular weight, e.g., methyltriphenylarsonium, give salts that have a very low water solubility is discussed in more detail. There are 34 references. N. E.

2037. A simple technique in paper disc chromatography. K. Lakshminarayanan (*Arch. Biochem. Biophys.*, 1954, **49** [2], 396-399).—Good separation of amino-acids is obtained on 7.5-cm filter-paper discs by using a fine capillary tube to control the rate of irrigation with the solvent (*n*-butanol-acetic acid - water, 4:1:5). W. H. C. SHAW

2038. Polarographic diffusion coefficients. C. L. Rulfs (*J. Amer. Chem. Soc.*, 1954, **76** [8], 2071-2074).—The determination by means of unstirred diaphragm cells of the polarographic diffusion coefficients (*D*) of  $\text{Ti}^{II}$ ,  $\text{Pb}^{II}$  and Cd in common supporting electrolytes is described. This method has practical utility in studies of diffusion-controlled uncharacterised polarographic processes as a means of more accurate estimation of polarographic *n* values than is possible by using approximate values for *D*. H. F. W. KIRKPATRICK

2039. Reduction of potassium permanganate in the presence of glass. M. J. Cotter (*Chem. & Ind.*, 1954, [15], 433).—It has been known for some time that solutions of  $\text{KMnO}_4$  are reduced to a certain extent in presence of glass. Here the influence of the surface area of the glass on the degree of reduction has been investigated. By carrying out the standard procedure for estimating carbonaceous matter in lake water, etc., with 100-ml aliquots of the same sample of water to which 60 g of ground glass of various particle sizes had been added, it was shown that the surface area of the glass is an important factor. Investigation into a possible quant. relation between the surface area and the extent of reduction is proceeding. J. M. JACOBS

See also Abstracts 2102, 2305.

## 2.—INORGANIC ANALYSIS

2040. Determination of tritium by ion-current measurement. K. E. Wilzbach, A. R. van Dyken and L. Kaplan (*Anal. Chem.*, 1954, **26** [5], 880-883).—Concn. of  $3 \times 10^{-10}$  to  $10^{-2}$  curie of  $^3\text{H}$  can be determined to within 1 per cent. by measuring the radiation-induced ion-current, *i*, with a vibrating-reed electrometer when the sample is introduced (in small amounts of carrier H) into an ionisation chamber (Borkowski or Brownell pattern, *Nucleonics*, 1952, **10**, 26) filled with methane, propane or hydrogen at approx. atm. pressure. The true value of *i* (after correction for background, ion-recombinations, and so on) is proportional to disintegration-rate, *r*, of  $^3\text{H}$ , and is independent of filling pressure and collecting voltage (180 to 450 V). Values of *i*  $< 10^{-13}$  amp. are measured by the rate of change of a  $12 \times 10^{-12}$ -farad capacitance within the electrometer, those  $> 10^{-13}$  amp. are measured by the voltage drop across a resistor. The charge collected per disintegration in methane, propane, and hydrogen has been determined by calibrating the ionisation-chamber with known amounts of  $^3\text{H}$ , thus permitting conversion of *i* to absolute values of *r*. The full procedure is described.

W. J. BAKER

2041. Dissolution of sodium-potassium alloys for purposes of analysis. J. C. White, C. K. Talbott and L. J. Brady (*Anal. Chem.*, 1954, **26** [5], 942-943).—The alloy sample (0.05 to 25 g) is immersed in *n*-hexane in a beaker resting in an oil-bath. A current of He or Ar is passed through a funnel inverted over the beaker (to exclude O) and pure methanol or ethanol is added dropwise, with occasional stirring to prevent formation of a coating over unreacted alloy. Finally, for safety, an excess of methanol is added followed by water to dissolve the alcoholate. The two phases are then transferred to a separating funnel and the heavier (water) layer (containing any metal impurities) is removed. Use of a plastic shield during the

operation is recommended; special care is needed if the alloy contains oxides, e.g.,  $\text{K}_2\text{O}$ , in which case the inert-gas filled dry-box technique is almost obligatory. W. J. BAKER

**2042. Quantitative separation of copper by thiourea from homogeneous solution.** S. Washizuka (*Bull. Chem. Soc., Japan.*, 1954, **27** [2], 76-79).—Separation of 10 to 100 mg of Cu from large amounts of Ca or Mg is effected by quant. pptn. as sulphide at  $100^\circ\text{C}$  and an initial pH of 4, using 1 to 2 ml of aq. 10 per cent. thiourea in presence of  $\approx 2\text{ g}$  of urea per 300 ml of soln. Boiling should be continued for 40 to 60 min. after the initial black turbidity forms, and the final pH should be  $\leq 8$ . The procedure is applicable also to soln. containing  $\geq 60\text{ mg}$  of Ni, 30 mg of Mn and 20 mg of Co or Zn, provided that 2 g of  $\text{NH}_4\text{Cl}$  are added to limit the final pH to 8. The well-washed and dried ppt. is ignited and weighed as  $\text{CuO}$ . W. J. BAKER

**2043. Spectrochemical determination of impurities in copper and copper alloys by means of a spark-ignited arc-like discharge.** F. V. Schatz (*Spectrochim. Acta*, 1954, **6** [3], 198-210).—The spectrometric determination of Pb, Sn, Fe, Si, Bi, Al ( $< 0.001$  per cent.), Te, As, P ( $< 0.004$  per cent.), and Zn ( $< 0.01$  per cent.) in Cu or Cu alloys is described in full. The power-circuit is adjusted to give a uni-directional heavily-overdamped lamellar discharge along a horizontal axis from the negative sample-plane to the pointed C anode, each pulse of this interrupted d.c. arc having a max. of 45 amp. and a duration of 6-3 milli-sec. Under these conditions a narrow zone of increased sensitivity for certain elements exists in the cathode region. Changes in the sensitivity of several elements and in the excitation temp. are studied at different points along the discharge gap (0 to 2.5 mm). Standard working curves for several possible analyses on Cu alloys are shown, the shifts caused by changes in concn. of the matrix element, presence of other elements, and changes in background intensity, being indicated. The method avoids the rapid build-up of the Cu spectrum in the normal spark-excitation procedure, besides permitting easy and accurate determination of 0.01 to 0.05 per cent. of P without background interference, since the 2535.6 line of Fe (interfering with the 2535.7 line of P) is suppressed at all Fe concn. of  $< 0.015$  per cent. W. J. BAKER

**2044. Simultaneous colorimetric determination of copper, cobalt and nickel as diethyldithiocarbamates.** J. M. Chilton (*Anal. Chem.*, 1954, **26** [5], 940).—Modified details, allowing the determination to be carried out in presence of  $\text{UO}_2^{++}$  and with increased sensitivity, of the method described in *Anal. Abstr.*, 1954, 24, are given. D. A. PANTONY

**2045. The spectrophotometric determination of magnesium with thiazol yellow dyes.** T. A. Mitchell (*Analyst*, 1954, **79**, 280-285).—The method for colorimetric determination of Mg by means of the red colour given with thiazol yellow is examined critically, and a method is proposed in which the Mg is pptd. as  $\text{Mg}(\text{NH}_4)_2\text{PO}_4$ , the ppt. is dissolved in acetic acid and the soln. is treated with a reagent containing the dye with glycerol as colour stabiliser and starch as protective colloid. It is finally made alkaline. The colour is measured spectrophotometrically at  $520\text{ m}\mu$  at a fixed time after its development. A calibration graph is prepared from soln. containing up to  $120\text{ }\mu\text{g}$  of Mg. Ca is

removed as oxalate in the initial stages of the method and Fe and Al are held in soln. by citrate in the  $\text{Mg}(\text{NH}_4)_2\text{PO}_4$  pptg. reagent. Up to  $50\text{ }\mu\text{g}$  of Mn in the aliquot taken do not interfere. A. O. JONES

**2046. Determination of magnesium and calcium in dolomite.** S. Shinkai (*J. Ceram. Ass. Japan*, 1953, **61**, 479).—The sample (0.2 g) is dissolved in HCl and is then treated with excess of 0.05 N  $\text{Ba}(\text{OH})_2$ ;  $\text{Ca}(\text{OH})_2$  remains soluble but the  $\text{Mg}(\text{OH})_2$  is pptd. Back titration with 0.05 N HCl indicates the amount of  $\text{Ba}(\text{OH})_2$ , from which figure the  $\text{MgCO}_3$  is calculated.  $\text{CaCO}_3$  is determined in a separate sample by  $\text{KMnO}_4$  titration. BRIT. CERAM. RES. ASS.

**2047. Removal of aluminium in the flame-photometric determination of calcium.** O. Gjems and D. Lydersen (*Z. Pflernähr. Düng.*, 1954, **64**, 36-41).—The extent of the interference of Al and Fe in flame-photometric determinations of Ca is examined. A portion of the test solution (such as soil extract), containing a total of  $\geq 6$  milliequivalents of  $\text{Al}^{+++}$ ,  $\text{Fe}^{+++}$  and  $\text{Cr}^{+++}$  is placed in a 100-ml graduated flask and neutralised to methyl red with acetic acid or aq.  $\text{NH}_3$ . To this is added 10 ml of a solution containing in 1 litre of glacial acetic acid (100 ml), aq. LiCl (6.1 g per litre) (250 ml) and  $\text{NH}_4\text{Cl}$  (100 g). For test solutions containing  $> 50\text{ mg}$  of Ca per litre the proportion of LiCl in this reagent must be increased. After mixing and dilution to the mark, an aliquot (50 ml) is heated with 10 ml of aq. ammonium benzoate (10 per cent.) in a covered beaker on a bath of boiling water for 30 min. After cooling and filtration, the soln. is suitable for the flame-photometric determination of Ca. A. G. POLLARD

**2048. Ethylmethyl picrate as a reagent for barium.** E. R. Caley and C. E. Moore (*Anal. Chem.*, 1954, **26** [5], 939-940).—A neutral soln. containing 2 mg/ml of  $\text{Ba}^{++}$  is treated with 15 ml of 3 per cent. aq. soln. of 3-ethyl-5-methyl-2:4:6-trinitrophenol sodium salt (pH 7). The ppt. is collected, washed with ether and dissolved in 50 ml of warm water; the volume is made up to 100 ml and the absorbancy is measured at  $390\text{ m}\mu$ .  $\text{Ba}^{++}$  concn. is derived from standards. Full details for the prep. of the reagent (nitration of 3-ethyl-5-methylphenol), and the light absorption properties of its Ba salt are given. D. A. PANTONY

**2049. A direct titrimetric determination of barium with ethylenediaminetetra-acetate.** R. Sijderius (*Anal. Chim. Acta*, 1954, **10** [6], 517-519).—In the titration of  $\text{Ba}^{++}$  with ethylenediaminetetra-acetic acid (**I**), the indistinct end-point can be improved by the addition of an Mg salt. The following procedure is recommended. To 100 ml of a soln. containing 3 to 110 mg of  $\text{BaCl}_2$ , add 1 ml of 2 N HCl and 4 ml of a buffer soln. made by dissolving 70 g of  $\text{NH}_4\text{Cl}$  in 1800 ml of aq.  $\text{NH}_3$  (25 per cent.) and adding a soln. of 10 g of the dipotassium magnesium salt of **I** in 200 ml of water; add 7 drops of a soln. of 0.5 g of Eriochrome black T (Solochrome black WFA) and 4.5 g of  $\text{NH}_2\text{OH}\cdot\text{HCl}$  in 100 ml of ethanol (95 per cent.) and titrate to a pure blue colour with a standard soln. containing 6.64 g of the disodium salt of **I** per litre. A soln. of **I** standardised against  $\text{CaCO}_3$  yields somewhat high results so it is best to standardise it against a Ba salt. W. C. JOHNSON

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**2050. Complexometric determination of barium sulphate.** P. J. Jackson (*Chem. & Ind.*, 1954, [15], 435).—Two points of technique are raised in connection with the previous communication of Belcher *et al.* (*Anal. Abstr.*, 1954, 1, 448). It is suggested that instead of using a pad of paper pulp, the  $\text{BaSO}_4$  should be filtered by suction through a disc of fine-textured filter-paper twice washed with acid and supported in a glazed porcelain Gooch crucible, and that triethanolamine should be used for the prep. of the Solochrome black indicator.

J. M. JACOBS

**2051. Contributions to the morphology of the barium sulphate precipitate produced by the method of L. W. Winkler.** E. Schulek, E. Pungor and F. Guba (*Anal. Chim. Acta*, 1954, 10 [6], 506-512).—Winkler ("Ausgewählte Untersuchungsverfahren für das chemische Laboratorium. Die chemische Analyse, XXIX," Ferdinand Enke, 1931, p. 93) recommended the pptn. of  $\text{BaSO}_4$  at pH 2 in the presence of  $\text{NH}_4\text{Cl}$ . Examination under the microscope and the electron microscope of crystals so produced indicates a regular rhombic structure with straight edges. Pptn. from neutral solutions or in the absence of  $\text{NH}_4\text{Cl}$  yields a cruciform structure with serrated edges. The poorer filtering qualities of the second type of ppt. are attributed to the brittleness of the projections.

W. C. JOHNSON

**2052. Recrystallisation of barium sulphate from fused salts.** M. Gallant, G. J. Schmitt and J. Steigman (*Anal. Chem.*, 1954, 26 [5], 846-849).—In order to avoid errors due to co-pptn. of extraneous ions ( $\text{Na}^+$ ,  $\text{Cl}^-$  and  $\text{NO}_3^-$ ),  $\text{BaSO}_4$  that has been pptd. by known methods is dissolved in 10 parts of fused (660° C eutectic)  $\text{KCl}$ - $\text{NaCl}$  mixture in a platinum dish. The cooled melt is leached with 100 to 200 ml of 2 per cent. aq.  $\text{BaCl}_2$  containing 0.5 per cent. of  $\text{HCl}$ . The residual  $\text{BaSO}_4$ , which is readily filtered, is ignited in the normal manner, and its wt. is found to agree closely with that expected from theory.

D. A. PANTONY

**2053. Distribution of strontium within barium sulphate precipitated from homogeneous solution.** L. Gordon, C. C. Reimer and B. P. Burr (*Anal. Chem.*, 1954, 26 [5], 842-846).—The heterogeneous distribution of  $\text{Sr}^{++}$  within  $\text{BaSO}_4$  obtained by "conventional" or methyl sulphate hydrolysis homogeneous pptn. methods is investigated by use of  $^{90}\text{Sr}$ . It is found that the homogeneous pptn. gives a ppt. of  $\text{BaSO}_4$  that has a smaller proportion of  $\text{Sr}^{++}$ , and that increased digestion time of the heterogeneously pptd.  $\text{BaSO}_4$  leads to greater co-pptn. of  $\text{Sr}^{++}$ . Evidence for the development of a supersaturated soln. of  $\text{BaSO}_4$  in the initial stages of pptn. is produced.

D. A. PANTONY

**2054. Determination of zinc by dithizone in a monophasic water-glycol system.** B. L. Vallee (*Anal. Chem.*, 1954, 26 [5], 914-917).—The sample (1.5 ml of blood plasma or serum treated with  $\text{HCl}$  and trichloroacetic acid and then centrifuged) is adjusted to pH 4 (thymol blue) with 6  $N$   $\text{HCl}$  or dil. aq.  $\text{NH}_3$  (1 + 1). Ammonium citrate soln. (25 per cent.) (0.2 ml at pH 4) and 2 ml of Zn-free buffer soln. (Na acetate -  $\text{Na}_2\text{S}_2\text{O}_3$  -  $\text{KCN}$  brought to pH 4 with acetic acid) are added and the volume is adjusted to 4 ml. 2-Methoxyethanol (3.5 ml) is added, the soln. cooled, and 0.01 per cent. dithizone in 2-methoxyethanol (0.5 ml) is thoroughly mixed in. The absorbancy of an aliquot is measured at 525  $m\mu$ , within 10 min. of mixing, with reference to a soln.

identical save for the absence of  $\text{Zn}^{++}$ . The Beer-Lambert law holds for the system, and mean deviation is  $\pm 2.1$  per cent. for aqueous solutions and  $\pm 6.6$  per cent. for blood plasma containing 3  $\mu\text{g}$  of  $\text{Zn}^{++}$ . Tolerance limits for several interfering ions are given;  $\text{Cu}^{++}$ ,  $\text{Cd}$ ,  $\text{Bi}$ ,  $\text{Ag}$ ,  $\text{Hg}^{++}$ ,  $\text{Ni}^{++}$  and  $\text{Co}^{++}$  interfere seriously.

D. A. PANTONY

**2055. Polarographic determination of zinc in alkaline zinc-plating solutions.** R. Diaz and E. H. Lindemann (*Plating*, 1953, 40 [7], 762-764).—Details are given of reagents, apparatus and procedure of a polarographic control method for the analysis of  $\text{Zn}$  in  $\text{Zn}$ -plating solution. Methods of eliminating interference by impurities  $\text{Cu}$ ,  $\text{Pb}$ ,  $\text{Cr}$ ,  $\text{Ni}$ ,  $\text{Co}$  are discussed, and results and precision of the measurements by the method are given.

METAL ABSTR.

**2056. Fluorimetric determination of cadmium.** N. Evcim and L. A. Reber (*Anal. Chem.*, 1954, 26 [5], 936-937).—To 25 to 50 ml of a soln. containing 0.1 to 2 mg of  $\text{Cd}^{++}$  is added an equal vol. of 95 per cent. ethanol and the mixture is warmed to 60° C. A slight excess of 0.1 per cent. 2-(*o*-hydroxyphenyl)benzoxazole in 95 per cent. ethanol is added and the pH is adjusted to 11 with aq.  $\text{NH}_3$  or  $\text{NaOH}$ . The ppt. is isolated after digestion, dried and then dissolved in 50 ml of glacial acetic acid. The fluorescence of this soln. is measured in a fluorimeter and the  $\text{Cd}^{++}$  concn. is determined from a calibration curve derived from standards treated in a manner similar to that described above. The  $\text{Cd}$  is determined to within  $\pm 0.02$  mg.

D. A. PANTONY

**2057. Volumetric precipitation methods of determining mercurous salts as  $\text{Hg}_2(\text{SCN})_2$ , using adsorption and oxy-adsorption indicators.** F. Burriel-Marti and S. Arribas Jimeno (*An. Soc. Esp. Fis. Quim.*, B, 1954, 50, 185-194). Various indicators have been compared for sharpness of end-point in the titration of mercurous salts with mercurithiocyanates, and *vice versa*. The best indicator system for the former titration was  $\text{Cu}^{++}$ -o-tolidine; for the reverse titration, bromophenol blue or  $\text{Fe}^{+++}$ -o-dianisidine was best. The influence of pH and concn. during the titration have been studied.  $\text{Zn}$  can be determined by pptn. with excess of mercurithiocyanate and titration of the excess with a mercurous salt.

L. A. O'NEILL

**2058. Photometric mercury analysis. Correction for organic substances.** A. E. Ballard, D. W. Stewart, W. O. Kamm and C. W. Zuehlke (*Anal. Chem.*, 1954, 26 [5], 921-922).—Errors caused by the presence of organic material in the determination of  $\text{Hg}$  by means of its absorption at 253.67  $m\mu$  (Zuehlke and Ballard, *Brit. Abstr. C*, 1950, 477) are avoided by measuring the absorbancy of the contents of the mercury photometer cell in a modified spectrophotometer at 253.7  $m\mu$ .  $\text{Hg}$  concn. is obtained by difference. D. A. PANTONY

**2059. Boron in sodium metal. Determination of microgram amounts by alcohol extractions.** J. Rynasiewicz, M. P. Sleeper and J. W. Ryan (*Anal. Chem.*, 1954, 26 [5], 935-936).—Na metal (2 g) containing 0.5 to 8.0  $\mu\text{g}$  of B is dissolved in 50 ml of  $\text{H}_2\text{O}$  under  $\text{N}_2$ , and the cooled soln. is almost neutralised with 4.5  $N$   $\text{HCl}$  (methyl red). After evaporation to dryness in Pt, the residue is made just acid with 10 per cent.  $\text{HCl}$  and the B is extracted with three 15-ml portions of 95 per cent. ethanol made just acid with  $\text{HCl}$ . The extracts are centrifuged and the

filtrate is neutralised with 0.1 N NaOH in a platinum dish. Then 5 ml of 0.1 N NaOH are added in excess, followed by 2 ml of 3 per cent. v/v glycerol in methanol. The mixture is evaporated to dryness and ignited; the residue is cooled in solid  $\text{CO}_2$  and 2 ml of 0.1 per cent. curcumin in 95 per cent. ethanol are added, followed by cautious addition of 6 per cent. w/v oxalic acid in dil. HCl (1 + 4). The mixture is evaporated to dryness at 60° C, and the residue is extracted with 70 per cent. ethanol, the extract being made up to 25 ml with the solvent. The soln. is centrifuged and the absorbancy of the supernatant liquid is measured at 550 m $\mu$ . Comparison is made with standards prepared by a similar procedure. All experiments are carried out in glassware of low B content. Experiments on the recovery of added B by this analytical procedure indicate  $72 \pm 16$  per cent. recovery of the element.

D. A. PANTONY

**2060. Determination of aluminium in iron and steel.** Methods of Analysis Committee (*J. Iron & Steel Inst.*, 1954, **176** [3], 263-267).—After mercury-cathode electrolysis, the Al is pptd. with ammonia. The ppt. is dissolved in HCl,  $\text{H}_2\text{SO}_4$  is added and the soln. is evaporated to fumes. A few drops of conc.  $\text{HNO}_3$  are added followed by  $\text{KMnO}_4$  soln. until the soln. is just pink; Ti, V, Fe, etc., are then removed with cupferron. The filtrate is fumed with conc.  $\text{HNO}_3$ , tartaric acid is added and the soln. is then neutralised with ammonia. KCN soln. is added, followed by ammonium sulphide. After filtration, 8-hydroxyquinoline is slowly added to the filtrate. To the resulting Al-complex dissolved in HCl, excess of standard  $\text{KBrO}_3$  soln. is added. After adding KI, the soln. is back-titrated with  $\text{Na}_2\text{S}_2\text{O}_3$  by use of starch as indicator. The thiosulphate is standardised against the  $\text{KBrO}_3$  (1 ml of  $\text{KBrO}_3 \equiv 0.0002$  g of Al). Results for synthetic solutions and steels are included.

C. J. KEATTCH

**2061. A study of the colorimetric determination of aluminium in steels.** M. Jean (*Anal. Chim. Acta*, 1954, **10** [6], 526-553).—A detailed investigation of the colorimetric determination of Al with stilbazo [ammonium 4:4'-di-(3:4-dihydroxyphenylazo)-stilbene-2:2'-disulphonate] in the presence of Fe is described. Ti and V yield colours with stilbazo, and their removal with various reagents is studied. Cr interferes and is removed by electrolysis, but Ni is without influence on the recommended methods that follow. *Steels containing > 0.1 per cent. of Al.*—Dissolve the steel in dil.  $\text{H}_2\text{SO}_4$ , oxidise it with  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  and electrolyse the soln. with the aid of a mercury cathode; neutralise with aq.  $\text{NH}_3$ , acidify with dil.  $\text{H}_2\text{SO}_4$ , add ascorbic acid to reduce and sequester Fe, adjust the pH to 5.5 with an acetate buffer, add an aq. soln. of stilbazo, and compare photometrically at 515 m $\mu$  with standards prepared by a similar procedure from pure Fe, known amounts of Al $^{+++}$  being added after the electrolysis. If such steels contain Ti and V, neutralise, after the electrolysis, with NaOH, acidify with dil.  $\text{H}_2\text{SO}_4$ , add cupferron and extract with  $\text{CHCl}_3$ . *Nitrided steels containing > 1.25 per cent. of Al.*—Dissolve the steel in dil.  $\text{H}_2\text{SO}_4$ , neutralise with aq.  $\text{NH}_3$ , acidify the soln. with dil.  $\text{H}_2\text{SO}_4$ , add ascorbic acid and proceed as above. When such steels contain Ti and > 2 per cent. of Al, electrolyse the soln. at a mercury electrode, neutralise with aq.  $\text{NH}_3$ , acidify with dil.  $\text{H}_2\text{SO}_4$  and ppt. the Ti with *p*-hydroxyphenylarsonic acid. *Magnetic steels containing > 15 per cent. of Al.*—

Use the same principle as for nitrided steels free from Ti. A polarographic examination of the reaction of VV with tungstosilicic acid confirms the formation of a tungstovanadosilicic complex.

W. C. JOHNSON

**2062. Methods for the spectrographic analysis of trace metals. IV. Spectrographic trace analysis of pure aluminium.** F. A. Pohl (*Z. anal. Chem.*, 1954, **142** [1], 19-27).—Two methods of trace analysis are described in which trace elements are separated by pptn. from the Al by means of Ti as carrier. Thioacetamide (I) precipitates Cd, Co, Cu, Fe, Mn, Ni, Pb, Zn, Ca, Cr, Mg and Ti in NaOH solution while ammonium tetramethylenedithiocarbamate (II) and thioglycolic acid, 2-naphthylamide (Thionalide) (III) precipitate Cd, Co, Cu, Fe, Ga, Mo, Ni, Sb, Sn, V, Zn, Cr, Mn and Pb completely and Ti partially in acetic acid solution.

*Method (a)*—Dissolve Al (1 g) in 15 per cent. NaOH, dissolving any residue in  $\text{HNO}_3$ , add 0.1 per cent.  $\text{Ti}^{\text{IV}}$  acetate or sulphate (3 ml) and 2 per cent. I (5 ml), boil for 3 min. and cool. Filter, wash ppt. with 1 per cent.  $\text{NH}_4\text{NO}_3$  soln. and dissolve it in a few drops  $\text{HNO}_3$ ; evaporate to dryness and transfer to carbon electrodes. *Method (b)*—Dissolve Al (1 g) in conc. HCl (10 to 15 ml), neutralise with aq.  $\text{NH}_3$  to methyl orange, add 0.1 per cent.  $\text{Ti}^{\text{IV}}$  acetate or sulphate (3 ml) and 25 per cent ammonium acetate buffer, pH 4, (20 ml), heat to 80° C and add in succession 5 per cent II (60 ml) and 5 per cent. methanolic III (5 ml); after 30 min., cool to 15° C and filter. Dissolve in a few drops  $\text{HNO}_3$  and transfer to electrodes. Iron if present in large quantity may be removed by extraction of the thiocyanate. Depending on their arc intensity, 0.01 to 0.00001 per cent. of the elements stated can be determined. P. S. STROSS

**2063. Spectrochemical analysis of aluminium alloys using molten metal electrodes.** L. D. Frederickson, jun., and J. R. Churchill (*Anal. Chem.*, 1954, **26** [5], 795-800).—A representative, but not necessarily uniform, sample of Al alloy is melted (700° to 1000° C) within a cratered graphite electrode by means of one of several methods (induction heating is preferred), and an arc is struck with a graphite counter-electrode. The spectrographic determination is completed in the normal manner. Precision is claimed to be at least equivalent to that obtained with uniform solid specimens.

D. A. PANTONY

**2064. The separation and determination of gallium.** G. W. C. Milner, A. J. Wood and J. L. Woodhead (*Analyst*, 1954, **79**, 272-279).— $\text{GaCl}_3$  is the halide of gallium most readily extracted from acid solutions by organic solvents. Several solvents, especially certain ketones, proved as effective as ether, but ether is preferable for the analysis of mixtures of Ga and U. After removal of solvent, the Ga is pptd. with camphoric acid from soln. buffered at pH 3.3 with a formic acid - ammonium formate buffer, the ppt. being collected in a sintered-glass crucible, dried and weighed. The factor for conversion of the wt. of ppt. to Ga is 0.213. For < 3-mg amounts of extracted Ga, determination is better accomplished by titration with  $\text{K}_4\text{Fe}(\text{CN})_6$  with 3:3'-dimethylnaphthidine as indicator.

A. O. JONES

**2065. The volumetric determination of ceric perchlorate.** T. Rigg (*Analyst*, 1954, **79**, 307-308).—The method for titration of  $\text{Ce}(\text{SO}_4)_2$  with  $\text{FeSO}_4$

with  $\text{Fe}^{II}$ -*o*-phenanthroline complex as indicator is not generally applicable to  $\text{Ce}(\text{ClO}_4)_4$  and was found useless for titration of a 0.001 *M* soln. The following procedure was found suitable. Excess of standard  $\text{FeSO}_4$  is run into the soln. and the excess is titrated with standard  $\text{Ce}(\text{SO}_4)_2$  soln., 2:2'-dipyridyl (0.5 g in 100 ml of conc. aq.  $\text{NH}_3$  and 100 ml of water) being used as indicator. The titration must be made in  $\approx M$  HCl. If  $\text{Fe}^{III}$  is present,  $\text{H}_3\text{PO}_4$  is added near the end-point to suppress the brownish  $\text{FeCl}_3$  complex.

A. O. JONES

**2066. Separation of rare earths by ion exchange.** **VII. Quantitative data for the elution of neodymium.** F. H. Spedding and J. E. Powell (*J. Amer. Chem. Soc.*, 1954, **76** [9], 2545-2549).—The elution of Nd from a Nalcite HCR bed with citric acid-ammonium citrate solution over a pH range 5.0 to 8.0 is described. A number of experimental difficulties encountered are mentioned. From the results, information is obtained about the movement of the bands and the distribution of the ions in the aq. and resin phases. The sum of the equiv. of Nd and  $\text{NH}_4^+$  in the resin is equal to the resin capacity, whilst the sum of the equiv. of Nd and  $\text{NH}_4^+$  in the aq. phase equals the  $[\text{NH}_4^+]$  in the eluant. Moreover—

$$\frac{[\text{Nd}]_{\text{resin}}}{[\text{Nd}]_{\text{aq}}} = \frac{[\text{NH}_4^+]_{\text{resin}}}{[\text{NH}_4^+]_{\text{eluate}}} = \frac{\text{capacity of resin}}{[\text{NH}_4^+]_{\text{eluant}}}$$

A nearly linear relationship is established between  $[\text{NH}_4^+]$  in the eluant and the sum of the  $[\text{NH}_4^+]$  and  $[\text{Nd}]$  in the eluate. The break-through vol. of the Nd, as calculated from dividing the number of equiv. of H-form resin remaining after the rare-earth band is adsorbed by the equiv. of  $\text{NH}_4^+$  per litre of eluant, is in excellent agreement with the observed values.

J. H. WATON

**2067. Separation of rare earths by ion exchange.** **VIII. Quantitative theory of the mechanism involved in elution by dilute citrate solutions.** F. H. Spedding and J. E. Powell (*J. Amer. Chem. Soc.*, 1954, **76** [9], 2550-2557).—The behaviour of rare earths on columns of cation-exchange resins is reproducible and the character of the bands obtained appears to result from the attainment of true equilibrium. By using classical thermodynamic relationships and the concepts of electrical neutrality and material balance, equations are obtained for the Nd-ammonium citrate exchange system. When substituted with experimental results, these equations lead to the evaluation of important constants of the system, so allowing theoretical curves to be constructed. The experimentally determined points from the Nd-ammonium citrate exchange system in the pH range 5.0 to 8.0 show excellent agreement with these curves. The results show that the free  $\text{Nd}^{III}$  in the eluate is negligible, being bound in citrate complexes of which the most important is  $[\text{Nd}(\text{citrate})_2]^{4-}$ .

J. H. WATON

**2068. Quantitative spectrochemical determination of praseodymium in lanthanum and europium in samarium on the medium dispersion spectrograph ISP-22.** G. Kryuger and R. R. Shvangiradze (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 11-21).—With copper electrodes, the lower one containing the rare-earth oxides mixed with four times their weight of  $\text{KHSO}_4$ , a Svenstitski a.c. activated arc and a medium dispersion spectrograph, satisfactory determinations are made of Pr (0.3 to 10 per cent.) in La and of Eu (0.02 to 10 per cent.) in Sm. The

comparison pairs of lines are Pr 390.8  $m\mu$  with La 393.62  $m\mu$ , and Eu 393.05  $m\mu$  with Sm 390.09  $m\mu$  for Eu up to 1 per cent. and Eu 281.4  $m\mu$  with Sm 279.68  $m\mu$  for Eu 0.1 to 10 per cent.

G. S. SMITH

**2069. Cupferron and neocupferron complexes of the rare-earth elements.** A. I. Popov and W. W. Wendlandt (*Anal. Chem.*, 1954, **26** [5], 883-886).—One g of rare-earth oxide is dissolved in 50 ml of 0.5 *N* HCl, and the cation is pptd. with an excess of 0.2 *M* cupferron or saturated neocupferron soln. The ppt. is collected by suction, dried at 110° C, weighed and ignited at 900° C. The effect of pH on the pptn. and the solubilities of the complexes is studied. Whereas the dried ppt. are not satisfactory for the determination of the rare earths, the ignited oxides are suitable, giving results that compare favourably with those by the oxalate method.

D. A. PANTONY

**2070. Conductimetric determination of carbon in metals.** J. E. Still, L. A. Dauncey and R. C. Chirnside (*Analyst*, 1954, **79**, 308).—In a paper on "Conductimetric Determination of Carbon in Metals" (*Anal. Abstr.*, 1954, **1**, 665), reference is made to a conductimetric apparatus devised by Gardner *et al.* (*Brit. Abstr. C*, 1950, 396) having, it is implied, a greased conical joint at the entrance to the combustion tube. This implication is now corrected, the joint being a dry joint with an accurately machined steel cone. The claim that an improvement had been effected in this particular is withdrawn.

A. O. JONES

**2071. Analytical methods for titanium and titanium alloys.** J. A. Corbett (*Metallurgia*, 1954, **49**, 206-208).—Methods used for the estimation of Fe, Mn, Al, Mg, Si, Cl, C, W, Mo, N and O when present as impurities in Ti, and for Fe, Mn, Cu, Co, Ni, Cr, Sn, Ta, Al and Ag, when occurring as binary alloys with Ti, are reviewed.

G. C. JONES

**2072. A field method for the determination of titanium in rocks.** L. Shapiro and W. W. Brannock (*Econ. Geol.*, 1953, **48**, 282).—About 4 mg of rock powder are fused with  $\text{KHSO}_4$  in a test-tube. Tiron (disodium 1:2-dihydroxybenzene-3:5-disulphonate) powder is added to the cool melt and the mixture is dissolved in a buffer solution. Sodium dithionite is added and the approx.  $\text{TiO}_2$  concn. is determined by comparing the resulting yellow solution with permanent standards. Results obtained by the method for  $\text{TiO}_2$  in 72 rock samples of various types are compared with those obtained by laboratory methods. *BRIT. CERAM. RES. ASS.*

**2073. A note to the article "Polarographic reduction of germanium" by A. K. Dasgupta and C. K. N. Nair.** P. Valenta and P. Zuman (*Anal. Chim. Acta*, 1954, **10** [6], 591-593).—Contrary to the statement of Dasgupta and Nair (*Anal. Abstr.*, 1954, **1**, 272) the second wave in the polarogram of Ge was noticed in the authors' previous paper (*Chem. Listy*, 1952, **46**, 478). The second wave is of a catalytic nature, and reasons are given for this conclusion. It is preferable to adopt conditions in which the second wave does not appear. The best results are obtained by using 0.1 *M* ethylenediaminetetraacetate buffered at pH 6 to 8.

W. C. JOHNSON

**2074. A rapid and accurate method for the determination of tin in tin-plate.** C. Courty (*Chim. et Ind.*, 1954, **71** [2], 270-272).—A piece of tin plate,

5 cm × 5 cm, is cut into 8 rectangles 1.25 cm × 2.5 cm. These are weighed and placed in a small glass receptacle with a sintered-glass base, which is then put in a 100-ml measuring cylinder containing 10 ml of HCl-SbCl<sub>3</sub> reagent (HCl, sp. gr. 1.1506, containing 20 g of SbCl<sub>3</sub> per litre). The cylinder is stoppered with a 2-holed rubber bung and a stream of CO<sub>2</sub> is passed. One minute after the rapid evolution of H<sub>2</sub> ceases, the glass receptacle is raised above the reagent level. The liquid is allowed to drain out and the receptacle is washed with HCl and then with water. The pieces of tin-plate are rubbed to remove deposited Sb and then dried and weighed; the loss in wt. gives an approximate figure for the amount of Sn on the tin-plate. For an accurate determination, the solution in the measuring cylinder is treated with excess of 0.1 N I and then back-titrated with 0.1 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> by using starch as indicator. 1 ml of 0.1 N I = 5.935 mg of Sn.

C. J. KEATTCH

**2075. Polarographic determination of lead in lead-plating solutions.** R. Diaz (*Plating*, 1953, **40** [3], 261-262).—A control method for the analysis of Pb in lead plating solutions is described. The diffusion current of the Pb ion in a medium containing KNO<sub>3</sub> forms the basis for the determination. The results agree with gravimetric values to within 2 per cent. for Pb concn. of 20 to 30 oz per U.S. gall.

METAL ABSTR.

**2076. The analytical chemistry of thorium. The use of sebacic acid and sodium naphthionate for its separation from uranium.** M. Nageswara Rao and Bh. S. V. Raghava Rao (*Z. anal. Chem.*, 1954, **142** [1], 27-30).—Sebacic acid as precipitating agent for Th (Smith and James, *J. Amer. Chem. Soc.*, 1912, **34**, 281) should only be used at a pH of 1.74 or higher; it will then separate Th from a seventy-fold excess of U. Sodium naphthionate, which is less useful, should only be used above pH 3.6. It will then separate Th from a ten-fold excess of U.

P. S. STROSS

**2077. Mass spectrometric determination of thorium.** G. R. Tilton, L. T. Aldrich and M. G. Inghram (*Anal. Chem.*, 1954, **26** [5], 894-898).—A standard soln. of <sup>230</sup>Th carrier is prepared and 100 μl (= 5 μg of Th) of this soln. are equilibrated with a soln. of the sample (preparation described). Enough pure Th for isotope analysis is then isolated from the equilibrated mixture by one or more of the following processes: (i) extraction at pH 2 into 0.1 M thenoyl-trifluoroacetone in benzene, (ii) extraction from an approx. saturated soln. of Al(NO<sub>3</sub>)<sub>3</sub> into isobutyl methyl ketone (giving 90 per cent. yield at each pass), or (iii) pptn. on La oxalate carrier. The final residue of Th(NO<sub>3</sub>)<sub>3</sub> is dissolved in 50 μl of 0.1 M HNO<sub>3</sub>, and half of the soln. is evaporated on a Ta ribbon (1 cm long, 0.001 × 0.025 in. cross-section), which is then loaded into a 6 or 12-in. 60° high-resolution mass spectrometer. The instrument should be equipped with an electron-multiplier giving an intensity-increase of 10<sup>4</sup>, so that isotopic analysis of 1 μg of Th is possible. Assuming absence of contamination (a blank is run in each analysis), the wt. (p.p.m.) of Th in sample (*w* g) is given by  $\frac{Th_s}{R_s} \left( \frac{R_c - R_s}{w R_s} \right)$ , where *R<sub>c</sub>* is <sup>230</sup>Th to <sup>232</sup>Th ratio of carrier, *R<sub>s</sub>* is that of carrier plus sample, and *Th<sub>s</sub>* is μg of Th in aliquot of carrier added. Data for granite, zircon, sphene and feldspar are reported and discussed briefly; the values are claimed to be accurate to ± 1.5 per cent. The procedure (3 to 5 working days required) is

invaluable where limited amounts of sample only are available or where Th concn. are abnormally low.

W. J. BAKER

**2078. Estimation of thorium by organic reagents. Part I. Phenylglycine-*o*-carboxylic acid.** S. K. Datta and G. Banerjee (*J. Indian Chem. Soc.*, 1954, **31** [2], 149-152).—A simple and quick method is described, which compares in accuracy with the *m*-nitrobenzoic acid method, involving reaction of excess of a boiling soln. of phenylglycine-*o*-carboxylic acid with a hot soln. of the thorium compound neutralised with NaOH to Congo red, separation of the ppt., and ignition to the oxide. The method is effective for separation of thorium from cerite earths.

M. TADMAN

**2079. A titrimetric method for total phosphoric anhydride.** J. T. R. Andrews (*J. Amer. Oil Chem. Soc.*, 1954, **31** [5], 192-195).—P<sub>2</sub>O<sub>5</sub> is determined in various phosphate mixtures, soap products and synthetic detergents by acid hydrolysis to the orthophosphate followed by potentiometric titration between the inflection points at pH 4.3 and pH 8.8. Heavy metals such as Fe, Al, Ca and Mg and buffering ions such as CO<sub>3</sub><sup>2-</sup>, SO<sub>3</sub><sup>2-</sup> and BO<sub>3</sub><sup>3-</sup> cause interference; SiO<sub>2</sub> must be removed during preparation of the sample if the ratio SiO<sub>2</sub> to P<sub>2</sub>O<sub>5</sub> ≥ 0.2. The method has been used for a wide range of routine experiments, the standard deviation for samples of synthetic detergents containing 25 to 30 per cent. of P<sub>2</sub>O<sub>5</sub> being ± 0.32.

N. M. WALLER

**2080. Determination of bismuth in pure bismuth-lead eutectic alloy. Improved phosphate method.** L. Silverman and M. Shideler (*Anal. Chem.*, 1954, **26** [5], 911-914).—The Bi-Pb alloy (1 g) is dissolved in 10 ml of dil. HNO<sub>3</sub> (1 + 3), and the soln. is evaporated nearly to dryness with 8 ml of 72 per cent. HClO<sub>4</sub>. After cooling and dilution with 30 ml of H<sub>2</sub>O, Pb<sup>2+</sup> (if > 0.2 g) is pptd. with 70 ml of dil. HCl (2 + 3). The PbCl<sub>2</sub> is filtered and washed free from Bi<sup>3+</sup> with 100 ml of dil. HCl (1 + 9). The soln. is further diluted with 100 ml of H<sub>2</sub>O and to it is added 50 ml of 6 per cent. cupferron. The Bi-cupferron ppt. is quickly filtered, washed and decomposed with dil. aq. NH<sub>3</sub> (1 + 19). The Bi ppt. remaining on the filter is dissolved in 25 ml of 70 per cent. HNO<sub>3</sub> and 15 ml of 72 per cent. HClO<sub>4</sub>. After decomposition of organic matter and evaporation nearly to dryness, the residue is cooled and diluted with 50 to 100 ml of H<sub>2</sub>O. The pH of the boiled soln. is adjusted to 0.6 with 70 per cent. HNO<sub>3</sub>, and 50 ml of phosphate reagent [31 g of (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> dissolved in a litre of water, with pH adjusted to 0.6 with HNO<sub>3</sub>] are added. The suspension is digested at 80° to 90° C for 0.5 hr, the ppt. is filtered, ignited at 650° C and weighed as BiPO<sub>4</sub>. The effect of pH and tolerance limits of Pb<sup>2+</sup> in the phosphate and cupferron precipitations are carefully examined; results are given for 4 Bi-Pb eutectic alloys. One per cent. of Fe has < 0.02 per cent. effect on the Bi<sup>3+</sup> determination.

D. A. PANTONY

**2081. Use of silver peroxide in volumetric and colorimetric determinations of vanadium.** M. Tanaka (*Bull. Chem. Soc. Japan*, 1954, **27** [1], 10-13).—In the volumetric method the sample (0.5 to 10 mg of V) is dissolved in dil. H<sub>2</sub>SO<sub>4</sub> (1 + 20), excess of H<sub>2</sub>O<sub>2</sub> is added to the filtered soln. and, after boiling and cooling, a suspension of Ag<sub>2</sub>O<sub>2</sub> in distilled water is added, the mixture being heated

for a few min. An excess of standard  $\text{FeSO}_4$  soln. plus 0.5 ml of 0.2 per cent.  $\alpha$ -phenanthroline are added to the cool soln., which is then titrated with  $\text{KMnO}_4$ . The error is  $\pm 0.01$  mg of V. Fe, Cr and (optionally) Mn should be first removed by electrolysis, at a mercury cathode. In the colorimetric method, the  $\text{Ag}_2\text{O}_2$  suspension is added to a soln. of the sample in 2 to 3 N  $\text{H}_2\text{SO}_4$ ; after heating for a few min. to form VV (adding oxalic acid if Mn is present), the soln. is divided into two parts. To a 10-ml aliquot of one part are added 1 ml of aq.  $\text{H}_3\text{PO}_4$  (1 + 2) and 0.5 ml of 5 M  $\text{Na}_2\text{WO}_4$ , the soln. is boiled a few min. and the colorimetric determination is made at  $410 \text{ m}\mu$  on the cool soln. in a cell 13 mm thick. Unless present in very small amounts K,  $\text{NH}_4^+$ , Ti, Zr, Bi, Sb and Sn should be removed first. The error is  $\pm 0.001$  mg on  $\geq 0.2$  mg of V. The methods are applicable to the analysis of V steels and can also be used to determine separately  $\text{V}^{\text{IV}}$  and  $\text{V}^{\text{V}}$ , e.g., when using ion-exchange resins for preliminary separation of V. W. J. BAKER

**2082. Quantitative analysis of niobium and tantalum in ores by fluorescent X-ray spectroscopy.** W. J. Campbell and H. F. Carl (*Anal. Chem.*, 1954, **26** [5], 800-805).—The technique of fluorescent X-ray spectroscopy is applied to the analysis of Nb and Ta ores in three possible ways. (a) The ore is decomposed by known methods and the mixed oxides are isolated by pptn. with tannin followed by ignition. These are packed into standard holders and exposed to a 35kV 35mA X-ray beam; the  $\text{NbK}_{\alpha}$  intensity is measured at  $15.25^\circ$  (NaCl crystal) by means of a Geiger counter, allowance being made for background intensity by subtraction of the count at  $16.2^\circ$ . The intensity of  $\text{TaL}_{\alpha}$  is measured in a 19kV beam at  $31.35^\circ$  and the background at  $33.2^\circ$  is subtracted. Intensities are converted to percentage compositions by means of calibration curves of  $\text{Nb}_2\text{O}_5$  in  $\text{Ta}_2\text{O}_5$  and  $\text{Ta}_2\text{O}_5$  in  $\text{Nb}_2\text{O}_5$  obtained at the two X-ray energy levels. A minimum concn. of  $\text{Ta}_2\text{O}_5$  of 1 to 2 per cent. is necessary for accurate determination; precision is said to be  $50 \pm 1$  per cent. or  $1 \pm 0.1$  per cent. of either oxide. (b) The mixed oxide sample containing an internal standard is made the target for a 50kV (Nb determinations) or 19kV 50mA (Ta) X-ray beam. The standards chosen are 10 per cent. of  $\text{MoO}_3$  or  $\text{ZrO}_2$  for  $\text{Nb}_2\text{O}_5$  determinations or 10 per cent. of  $\text{HfO}_2$ ,  $\text{WO}_3$ ,  $\text{ZnO}$  or  $\text{CuO}$  for  $\text{Ta}_2\text{O}_5$  determinations; intensities of  $\text{NbK}_{\alpha}$  and  $\text{TaL}_{\alpha}$  are measured as in method (a), and allowance is made for background, with reference to the standards'  $K_{\alpha}$  or  $L_{\alpha}$  spectral lines. The percentage composition is derived from calibration curves. Up to 15 to 20 samples per day, with a precision of  $\pm 5$  per cent. provided that  $> 0.01$  per cent. of  $\text{Nb}_2\text{O}_5$  or  $> 0.03$  per cent. of  $\text{Ta}_2\text{O}_5$  are present, may be analysed. For a limited number of determinations, a modification requiring an approx. 1 to 1 ratio of  $\text{Nb}_2\text{O}_5$  or  $\text{Ta}_2\text{O}_5$  to reference standard oxide is described. (c) For samples containing 0 to 5 per cent. of mixed oxides the linear relationship between intensity of spectral lines and concentration is used. The sample is used as the X-ray target, and  $I_{\text{NbK}_{\alpha}}$  and  $I_{\text{TaL}_{\alpha}}$  are determined as in method (b). A small known addition of  $\text{Nb}_2\text{O}_5$  or  $\text{Ta}_2\text{O}_5$  is made, and the increased intensities are measured; the concn. of  $\text{Nb}_2\text{O}_5$  and  $\text{Ta}_2\text{O}_5$  are calculated from a linear equation. Precision is given as  $0.5 \pm 0.05$  per cent. or  $10 \pm 0.5$  per cent. for both oxides. The relative merits of the three methods are discussed in relation to analytical requirements. D. A. PANTONY

**2083. Spot test for sulphides.** P. K. Mueller and M. C. Rand (*Water & Sewage Wks.*, 1954, **101** [5, Pt. 2], r 208).—Raschig's azide-iodine reaction, developed as a spot-test by Feigl, can be used to detect sulphides in water, sewage and industrial wastes. About 1 ml of the sample is placed in a watch-glass on a dark background. About 1 ml of 30 per cent. Na azide and about 1 ml of 0.1 N I are added. Bubbles of N form on the surface of the glass within 2 to 5 min. if  $\text{S}^{2-}$  is present.  $\text{S}_2\text{O}_3^{2-}$  and  $\text{CNS}^-$  interfere, but  $\text{SO}_4^{2-}$ ,  $\text{SO}_3^{2-}$  and elemental S do not react. Concn. of 0.1 p.p.m. can be easily detected. A. J. MEE

**2084. Assessment of individual sources of error in the combustion method for the determination of sulphur in steel. I. Mild steel.** T. B. Smith, A. Backhouse and P. Woodward (*J. Appl. Chem.*, 1954, **4** [2], 75-82).—Under the conditions normally realised industrially, only 86 per cent. of the S in the mild steel (S content = 0.048 per cent.) is liberated as  $\text{SO}_2$  at  $1300^\circ \text{C}$ . Of the remaining 14 per cent.,  $\approx 6$  per cent. is retained in the residue, and 8 per cent. is converted to  $\text{SO}_3$ . Three per cent. of the  $\text{SO}_3$  is carried over with the  $\text{SO}_2$ , giving an 89 per cent. recovery of the S if the estimation is completed acidimetrically with  $\text{H}_2\text{O}_2$  and standard  $\text{NaBO}_2$  instead of iodimetrically as before. Four per cent. of the  $\text{SO}_3$  condenses in the apparatus, and 1 per cent. escapes absorption. If a reduced-end combustion tube is used and the cooler parts of the train are rinsed before titration, the yield of 89 per cent. can be increased to 92 per cent. with a 2 per cent. loss of S as  $\text{SO}_3$ . For the best results, the ignition temp. should be  $1350^\circ$  to  $1400^\circ \text{C}$ , the time of pre-heating 3 min., the rate of O flow 3 litres per min., and the 1 g of sample should be spread evenly. Reduction of the amount of sample to 0.5 g gives only a 1 per cent. improvement. J. H. WATON

**2085. Study of the ether extraction of chromium in the form of perchromic acid.** K. V. Troitsky (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 51-55).—The influence of the nature of the acid ( $\text{HCl}$ ,  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ ), the acid concn. and the  $\text{H}_2\text{O}_2$  concn. are studied. Best results in the extraction of small amounts of Cr ( $33.3 \mu\text{g}$  per ml) are obtained with  $\text{HCl}$ . The concn. of  $\text{HCl}$  and  $\text{H}_2\text{O}_2$  must be low ( $0.1$  to  $0.5$  per cent.). G. S. SMITH

**2086. Determination of the sulphate content of chromates and dichromates.** H. Wolf and W. Petzold (*Z. anal. Chem.*, 1954, **141** [6], 429-433).—When  $\text{BaCl}_2$  is added to a solution having a chromate to sulphate ratio of more than 10:1, co-precipitation occurs even in acid solution. Hence, excess of chromate is first removed by pptn. with  $\text{HClO}_4$ . To 2 g of  $\text{K}_2\text{CrO}_4$  in 10 ml of water, add 70 per cent.  $\text{HClO}_4$  (15 ml) and heat on a water-bath until a white cloudiness appears. Filter, wash the pptd.  $\text{CrO}_3$  with  $\text{HClO}_4$ , dilute the combined filtrate and washings with water (100 ml) and neutralise with  $\text{NaOH}$ . Re-acidify with 2 N  $\text{HCl}$  (3 ml), add  $\text{BaCl}_2$ , filter and weigh the pptd.  $\text{BaSO}_4$  or measure the turbidity. P. S. STROSS

**2087. Analysis of uranium-zinc alloys.** J. S. Fritz, M. O. Fulda, S. L. Margerum and E. I. Lane (*Anal. Chim. Acta*, 1954, **10** [6], 513-516).—Methods are presented for the determination of Zn and U in Zn-U alloys containing up to 20 per cent. of U.

*Procedure for Zn*—Dissolve 0.4 g of the alloy in 10 ml of conc.  $\text{HCl}$  with the aid of  $\text{H}_2\text{O}_2$  and dilute

to 100 ml. Dilute 5 ml to 70 ml and add 10 ml of 0.05 *M* disodium ethylenediaminetetra-acetate (I). Add  $\text{NaHCO}_3$  (2 to 3 g) to produce a clear soln. and adjust to pH 8 to 8.5 with dil. HCl or 10 per cent. aq. NaOH. Titrate the excess of I with 0.05 *M*  $\text{ZnCl}_2$ , using 5 to 10 drops of 1 per cent. aq. ammonium purpurate (murexide) as indicator. The indicator blank is equivalent to 0.02 ml of 0.05 *M*  $\text{ZnCl}_2$ . *Procedure for U*—Take an aliquot of the original soln. containing  $\approx 25$  mg of U and dilute it to 80 ml with 3 *N* HCl. Pass this soln. through a lead reductor into *N* ferric alum and titrate with 0.01 *N* ceric sulphate using ferroin as indicator. W. C. JOHNSON

**2088. Gravimetric determination of fluorine as lanthanum fluoride.** A. I. Popov and G. E. Knudson (*Anal. Chem.*, 1954, **26** [5], 892-894).—Ten ml of 70 per cent.  $\text{HClO}_4$  are mixed with a sample containing 10 to 100 mg of F'. The mixture is distilled at 135° to 155° C in the presence of soda-glass beads; water is added slowly during the distillation of 175 to 200 ml (40 to 50 min.). Ten g of  $\text{NH}_4\text{NO}_3$  are added to the distillate, which is made alkaline with aq.  $\text{NH}_3$  (methyl orange), and then acid with  $\text{HNO}_3$ . A known excess of 2.5 per cent.  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  is added to the warm soln., followed by 0.5 ml of glacial acetic acid; aq.  $\text{NH}_3$  (3 *N*) is then added until pptn. is complete. The excess of La in the filtrate and washings, obtained after filtration or, preferably, centrifuging, is determined by a standard gravimetric procedure involving the ignition of a cupferron ppt. Formulae are given for calculation of F' in the original sample. Determinations of F' in NaF and  $\text{CaF}_2$  show average errors of 0.50 and 0.79 per cent., respectively. Figures are also given for the analysis of sodium monofluorophosphate, and a modified procedure is described for the determination of F in *p*-fluoroacetanilide. In these two determinations agreement with theory is good. D. A. PANTONY

**2089. The polarographic determination of fluoride. II. The determination of fluorine in bromine, hydrochloric acid and hydrobromic acid.** J. S. Beveridge, B. J. MacNulty, G. F. Reynolds and E. A. Terry (*Analyst*, 1954, **79**, 267-272).—The application of the method described in Part I (*Anal. Abstr.*, 1954, **1**, 1838) to the determination of F in Br, HCl and HBr is described. Results are satisfactory, particularly those for HCl and Br, but for HBr there appears to be a factor causing a wider spread of results. In general, and particularly with HBr, the cathode-ray polarograph appears to be superior to the conventional instrument. A. O. JONES

**2090. Separation of fluorine by distillation according to Willard and Winter.** G. Brunisholz and J. Michod (*Helv. Chim. Acta*, 1954, **37** [3], 874-878).—An automatically controlled constant-temp. still for the Willard-Winter separation of F is illustrated and described. The troublesome double distillation procedure commonly used in the presence of  $\text{PO}_4^{3-}$ , Al and Fe is avoided. Distillation is effected with  $\text{H}_3\text{PO}_4$  instead of  $\text{HClO}_4$ . H. WREN

**2091. Determination of fluoride. Procedure based upon diffusion of hydrogen fluoride.** L. Singer and W. D. Armstrong (*Anal. Chem.*, 1954, **26** [5], 904-906).—A 50-ml diffusion bottle is improvised from a polythene bottle, and in it is placed up to 1 ml of F'-containing soln. The apparatus is cooled to  $< 0^\circ \text{C}$ , 2 ml of 70 per cent.  $\text{HClO}_4$  are injected into it and a NaOH-coated polythene strip is inserted. The closed bottle is incubated at 50° C for 20 hr. and

cooled, and the NaF on the receiving strip is collected in water. The F' is determined by standard methods. Errors are  $\approx \pm 2$  per cent.  $\text{Na}_2\text{CO}_3$ , urea, glucose,  $\text{Al}_2\text{O}_3$ , NaCl and  $\text{Ca}_3(\text{PO}_4)_2$  do not interfere unless in  $> 250$ -fold excess.

D. A. PANTONY

**2092. Determination of water in easily hydrolysed fluorides.** J. G. Feibig and J. C. Warf (*Anal. Chem.*, 1954, **26** [5], 927-928).—Fluorides of U, Th ("dry" or as hydrates) or Be (1 to 10 g) in a nickel boat are placed in a nickel tube at 500° C under a N stream. The HF is trapped in a nickel tube packed with coarsely crushed  $\text{Na}_2\text{CO}_3$  at 300° C, and the issuing water is collected in  $\text{Mg}(\text{ClO}_4)_2$  and determined gravimetrically. Close agreement with theoretical water contents of the fluorides is attained.

D. A. PANTONY

**2093. High-frequency titration of micro quantities of chloride and sulphate.** G. S. Bien (*Anal. Chem.*, 1954, **26** [5], 909-911).—The sample (0.3 ml) containing  $\approx 5$  mg of  $\text{Cl}^-$  and 1 mg of  $\text{SO}_4^{2-}$  is made acid with 0.1 *N* acetic acid (methyl red), boiled and diluted with 40 ml of dioxan. The soln. is made up to  $\approx 100$  ml with water in a titration cell incorporated into the circuit of an oscilloscope. Ba acetate, standardised against  $\text{H}_2\text{SO}_4$ , and Ag acetate, standardised against KCl, are run in successively. The end-points are marked by minima of the titration curves relating dielectric constant to vol. of reagent. Minimum concn. that can be determined are:  $\text{Cl}^-$ , 0.5 mg;  $\text{SO}_4^{2-}$ , 0.15 mg. The precision claimed is  $\approx \pm 2$  per cent.

D. A. PANTONY

**2094. Copper absorbent for halogens and hydrogen sulphide in their determination in air.** P. M. Sadovsky (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 58-59).—Complete absorption of halogens and hydrogen sulphide occurs when air containing them is passed [rate of flow not stated] through a tube containing pure copper powder. G. S. SMITH

**2095. Colorimetric determination of iron by means of ethyl  $\alpha$ -isonitrosoacetate.** A. Boucherle (*Ann. Pharm. Franc.*, 1953, **11** [7-8], 540-546).—A 2 per cent. aq. soln. of ethyl  $\alpha$ -isonitrosoacetate gives a blue coloration with  $\text{Fe}^{II}$  salts in a soln. buffered with boric acid-borate at pH 7.8 at concn. as low as 1:500,000. The coloration reaches a max. in 5 min. and is stable for 7 to 8 hr. The method is specific, errors being  $> 5$  per cent. in the presence of Zn, Mg and Al and  $< 2$  per cent. in the presence of Ni or Cu.  $\text{Fe}^{III}$  may be determined after reduction with hydrazine. The  $\text{Fe}^{II}$  complex is of use as an indicator in oxidation-reduction reactions. N. M. WALLER

**2096. Lecture bench demonstration of the Zimmermann-Reinhardt method for titration of iron.** H. Schäfer (*Angew. Chem.*, 1954, **66** [8], 229-230).—The inhibition by  $\text{MnSO}_4$  of the oxidation of  $\text{Cl}^-$  by  $\text{KMnO}_4$  in the titration of  $\text{Fe}^{II}$  can be demonstrated by passing N through: (a) a solution containing only  $\text{KMnO}_4$  and HCl; (b) as (a) plus  $\text{FeSO}_4$ ; (c) as (b) plus  $\text{MnSO}_4$ . Only in (b) is Cl produced (revealed by the liberation of I from KI through which the N is subsequently passed).  $\text{Fe}^{III}$  instead of  $\text{Fe}^{II}$  produces no Cl; the effect of  $\text{Fe}^{II}$  must be due to formation of an unstable compound containing Fe with an oxidation number  $> 3$  that oxidises  $\text{Cl}^-$  or  $\text{Mn}^{II}$  preferentially if present, and not to reduction of the redox potential for the reaction  $\text{MnO}_4^- + 8\text{H}^+ + 5e \rightarrow \text{Mn}^{2+} + 4\text{H}_2\text{O}$ , as is sometimes claimed. R. C. MURRAY

**2097. Selective reaction for cobalt.** G. S. Goldberg (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 56-57).—The thiourea reaction of Rosenheim *et al.* (*Z. anorg. Chem.*, 1906, **49**, 13) is studied. By grinding solid  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  diluted with  $\text{NaNO}_3$  with thiourea, a blue or green colour is obtained (min. amount detectable is 0.31  $\mu\text{g}$ , limiting concn. 1:460,000). By boiling 1 ml of a Co solution with 1 ml of saturated thiourea solution, immersing a filter-paper in the mixture and then drying the paper a green colour appears (2.5  $\mu\text{g}$  of Co, limiting concn. 1:20,000). Bi, Cu, Ni and Cr interfere.

G. S. SMITH

**2098. Use of ion-exchange resins in micro-analysis. I. Detection of very small amounts of cobalt by means of ammonium thiocyanate.** M. Fujimoto (*Bull. Chem. Soc. Japan*, 1954, **27** [1], 48-50).—A micro-colorimetric procedure for detecting a min. of 0.16  $\mu\text{g}$ , or concn. of 1 in  $1.3 \times 10^5$ , of Co in soln. by using colourless or pale-yellow grains of Amberlite IRA 400 is described. One drop of sample (neutral or acid soln.) is placed on a white spot-plate with a few grains of the resin; on mixing with one drop of  $\text{M NH}_4\text{CNS}$  there is developed after a period varying from some min. to 24 hr. (depending on Co concn.) a light-blue coloration around the edges of, or throughout, the resin grains. The test is highly sensitive and the reaction is unaffected by presence of  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{UO}_2^{2+}$ ,  $\text{OH}^-$ ,  $\text{CN}^-$ ,  $\text{F}^-$ ,  $\text{I}^-$ ,  $\text{S}_2\text{O}_8^{2-}$ , and most acids, including acetic, oxalic, citric and tartaric.

W. J. BAKER

**2099. Rapid volumetric method for the estimation of nickel.** S. D. Pishawikar and D. G. Pishawikar (*Curr. Sci.*, 1954, **23** [3], 99).—The nickel solution (0.5 per cent. aq.  $\text{NiSO}_4$  or 0.2 per cent. aq.  $\text{NiCl}_2$ ) is treated with aq.  $\text{NH}_3$  (to a turbid or blue solution), acidified with acetic acid and titrated with standard dimethylglyoxime soln. The end-point is determined by filtering a drop of the soln. and testing the eluate on filter-paper with dimethylglyoxime solution. An accurate determination is then made by adding the dimethylglyoxime soln. to the nickel soln. to within 0.5 ml of the end-point, heating (water-bath) for 3 min. and completing the titration. The method is accurate and unaffected by the presence of Ag, Zn, Fe and Cr, but cannot be used when Cu or Co are present.

D. BAILEY

**2100. Proposed methods for chemical analysis of electronic nickel.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 160-164).—The methods are applicable to electronic Ni containing Co, Cu or  $\text{Fe}$  (0.01 to 1 per cent.), Ti (0.001 to 0.1 per cent.), and Mn (0.01 to 1.5 per cent.). The Co is determined by adding 0.75 per cent. aq. nitroso-R salt to a hot  $\text{HNO}_3$  solution of the metal buffered with Na acetate and then taking a photometric reading of 520  $\text{m}\mu$ . The elements usually present in electronic Ni do not interfere if they are at concn. less than the max. limits indicated above. Copper is determined at 600  $\text{m}\mu$  by photometric measurement of the violet-coloured complex in  $\text{HBr}-\text{H}_3\text{PO}_4$ . Noble metals other than Ag interfere. Mn is oxidised to  $\text{KMnO}_4$  by means of  $\text{KIO}_4$ , and its concn. is measured photometrically at 515  $\text{m}\mu$ . Reducing substances must be removed or destroyed before oxidation with  $\text{KIO}_4$ . The Fe and Ti are separated from other elements by pptn. with aq.  $\text{NH}_3$  and the Tiron (disodium 1:2-dihydroxybenzene-3:5-disulphonate) complex with Fe is measured at

560  $\text{m}\mu$ . The solution is then bleached with sodium dithionite and the Ti is measured at 390  $\text{m}\mu$ .

J. M. JACOBS

**2101. A new photometric determination of palladium with dimethylglyoxime.** W. Nielsch (*Z. anal. Chem.*, 1954, **142** [1], 30-35).—Pd-dimethylglyoxime is soluble in chloroform and can thus be separated from Au and Pt and directly estimated. Dissolve the sample in aqua regia and dilute suitably; to an aliquot, add 0.5 per cent. methanolic dimethylglyoxime (2 ml), shake for 15 sec. and extract 3 times with chloroform (10 ml). To the chloroform soln., add anhyd.  $\text{Na}_2\text{SO}_4$  (2 g) and measure the absorption at 350 to 360  $\text{m}\mu$ . The Beer-Lambert law is obeyed, and from 16 to 160  $\mu\text{g}$  of Pd can be accurately estimated.

P. S. STROSS

**2102. Gas chromatography. II. Separation and analysis of gas mixtures by chromatographic methods.** N. H. Ray (*J. Appl. Chem.*, 1954, **4** [2], 82-85).—Chromatographic methods for gas analysis by elution development are described. This technique is more rapid than that of displacement development or fractional desorption by heat, although the accuracy may be lower. Differentiation between the compounds is better, and the same column packing may be used for a large number of analyses. In a gas-liquid partition column with a "dinonyl" phthalate as the stationary phase (*Anal. Abstr.*, 1954, **1**, 955), the heavier gases such as propane, propylene and the  $\text{C}_4$  hydrocarbons can be separated at room temp. A column packed with active charcoal is used to separate the lighter gases such as  $\text{H}_2$ ,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{CH}_4$ ,  $\text{C}_2\text{H}_4$  and  $\text{C}_2\text{H}_6$  at temp. of 20° to 40° C.

J. H. WATON

**2103. Cornish stone. Quantitative mineralogical analysis.** P. S. Keeling (*Trans. Brit. Ceram. Soc.*, 1954, **53** [1], 67-75).—A thin section of the stone is prepared and the various minerals are distinguished by staining after suitable etching. The slide is carried on a modified mechanical stage, which moves the microscope cross-wires small fixed distances, and the number of times a particular mineral appears under the wires during several traverses of the section is recorded on a hand-operated electrical counter. A count of 1400 points is taken at distances of 0.3 mm along lines 0.8 mm apart. The first count covers quartz, Na and K feldspars, mica and kaolinite, and a second count covers topaz, apatite, fluorspar and tourmaline. Quartz is unstained, and, before the section is etched, staining with methylene blue distinguishes mica (pale blue) and kaolinite (dark blue). After etching with 40 per cent. aq. HF at 50° C (in a polythene container) and staining with 50 per cent. aq. Na cobaltinitrite (below room temp.), the K feldspar is bright yellow and the Na feldspar is unstained. Secondary mica is identified by its form of small plates and fan-shaped crystals in and around the edges of feldspar or as a microcrystalline mass, fluorspar by its mauve to purple colour and tourmaline by its greenish colour, pleochroism and shape. Primary tourmaline occurs as large eroded crystals and secondary tourmaline as radiating needles. Topaz and apatite are not separated.

J. A. SUGDEN

**2104. Cement content determination and reactive aggregate. Co-operative laboratory tests.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 282-285).—An investigation into the extent, if any, to which the inclusion

of an aggregate of known chemical activity might affect cement content determinations by A.S.T.M. method C85-42 (based on silica content) is reported. Determinations of the CaO content of the solutions obtained by this procedure were also made. The results were in good agreement and the average values found were close to the known amounts of sol.  $\text{SiO}_2$  and sol. CaO in the components of the concrete specimens. When allowance is made for the  $\text{SiO}_2$  and CaO dissolved from the aggregates and additives, the cement content determined is slightly higher than (but reasonably close to) the true cement content, except for diabase trap rock aggregate.

J. M. JACOBS

**2105. Significance of boiler deposit analysis.** F. E. Clarke and R. D. Hopkins (*Ind. Eng. Chem.*, 1954, **46** [5], 979-982).—Examples of problems encountered in boiler work are quoted, which stress the need for a thorough analysis of unusual deposits. Chemical analysis should always be accompanied by a detailed examination of the deposit structure.

J. H. WATON

See also Abstracts 2038, 2182, 2183, 2256, 2257, 2258, 2297.

### 3.—ORGANIC ANALYSIS

**2106. Determination of carbon and hydrogen by calorimeter bomb.** R. K. S. Mehta (*J. Sci. Ind. Res.*, B, India, 1954, **13** [3], 195-203).—A method is described for determining C and H with accuracies of  $\pm 0.15$  per cent. and  $\pm 0.05$  per cent., respectively, by means of a calorimeter bomb. Allowance is made for the effects of S and N, and it is possible to determine both C and H in one experiment if the bomb is quite dry. The method is especially useful for the analysis of volatile fuels, but is suitable for all fuels.

G. C. JONES

**2107. Replacement of lead peroxide in carbon-hydrogen micro-determination.** C. K. Cross and G. F. Wright (*Anal. Chem.*, 1954, **26** [5], 886-890).—Trishydroxylamine phosphate and sulphamic acid are used for absorption of N oxides in C and H determinations in compounds containing up to 50 per cent. of N. Results are superior to those obtained when  $\text{PbO}_2$  is used as absorbent, and the hydroxylamine salt is preferred because of its longer effective life.

D. A. PANTONY

**2108. Determination of epoxides with sodium sulphite.** J. D. Swan (*Anal. Chem.*, 1954, **26** [5], 878-880).—The sample, containing 0.01 to 0.02 mole of epoxide, is added to 50 ml of saturated  $\text{Na}_2\text{SO}_3$ . After agitation for 0.5 to 3 hr., the soln. is titrated with standard 0.2 N HCl with mixed alizarin-yellow R-xylene cyanol FF as indicator. A precision of  $\pm 0.5$  per cent. is claimed.

D. A. PANTONY

**2109. The analysis of combustion products. II. The estimation of traces of methanol in the presence of formaldehyde.** Sir A. C. Egerton, G. J. Minkoff and K. C. Salooja (*Anal. Chim. Acta*, 1954, **10** [6], 523-525).—Methanol in soln. is separated from a large excess of formaldehyde and of  $\text{H}_2\text{O}_2$  by treating the soln. with  $\text{NaHSO}_3$ , placing a drop on filter-paper and using ether as a chromatographic solvent. The methanol moves with the solvent front, the formaldehyde and  $\text{H}_2\text{O}_2$  remain as a max. at the point of application. The paper is cut into a number of portions and these are extracted

with water. Any methanol present is determined by oxidation with  $\text{KMnO}_4$  to formaldehyde and spectrophotometric determination of the formaldehyde by the chromotropic acid method of Boos (*Brit. Abstr. C*, 1949, 113). When no methanol is present the formaldehyde content falls off regularly from the point of application; the presence of methanol is shown by a steep rise towards the solvent front.

W. C. JOHNSON

**2110. Analysis data for the ternary system isopropanol - isopropyl ether - water.** W. S. Brey, jun. (*Anal. Chem.*, 1954, **26** [5], 838-842).—Refractive index, density and viscosity of various isopropanol-isopropyl ether-water soln. were measured at 25°C. From these data the ranges of composition over which various combinations of the physical properties are best suited for analytical purposes are indicated.

G. P. COOK

**2111. Semi-micro-determination of acetylation equivalents of alcohols.** R. E. Kepner and A. D. Webb (*Anal. Chem.*, 1954, **26** [5], 925-927).—The method is a modification of the techniques of Smith and Bryant (*J. Amer. Chem. Soc.*, 1935, **57**, 61) and Kaufmann (*Ber.*, 1937, **70B**, 2549). The pyridine is omitted, the alcohol, acetyl chloride and toluene mixture being refluxed for 1 hr. in a flask fitted with an internal cold-finger condenser. The flask is vented through a trap containing standard NaOH in order to prevent loss of acid. A modified reaction flask, enabling samples from 10 to 20 mg to be determined, is also described. The precision and accuracy for these sample wt. are within 2 per cent.

G. P. COOK

**2112. Quantitative [mass-spectrometric] analysis of some mixtures of alcohols and fatty acids.** F. Hageman and J. van Katwijk (*Ind. Chim. Belge*, 1954, **19** [4], 391-394).—Satisfactory results have been attained in the analysis of known mixtures of: (i) methanol, ethanol and  $\text{H}_2\text{O}$ ; (ii) methanol, ethanol, *n*-propanol, isopropanol and water and (iii) 9 carboxylic acids, by use of the Metro-Vickers mass-spectrometer.

R. C. MURRAY

**2113. Determination of 1:2-propylene glycol in ethylene glycol.** I. M. Baumel (*Anal. Chem.*, 1954, **26** [5], 930-931).—The glycols are oxidised with periodic acid, the resulting aldehydes are distilled and acetaldehyde is determined from its absorbance at 277 m $\mu$ . The calibration curve is prepared by oxidising known quant. of 1:2-propylene glycol and proceeding as for the sample. The error is  $\pm 0.22$  per cent.

G. P. COOK

**2114. The determination of small amounts of trimethylene glycol in high gravity and chemically pure glycerin.** W. D. Pohle and S. E. Tierney (*J. Amer. Oil Chem. Soc.*, 1954, **31** [5], 203-204).—A procedure applicable to pure and high gravity glycerol for the determination of trimethylene glycol and similar constituents is described. The percentage of water (a) is determined by the Fisher volumetric method and the sp. gr. of the sample at 25°C is measured (b). The sp. gr. at 25°C of a glycerol-water soln. containing (100 - a) per cent. of glycerol is determined (d) and the percentage of trimethyleneglycol is calculated from  $\frac{(d-b)}{0.0023}$  where 0.0023 is the sp. gr. change caused by a change in trimethylene glycol content of 1 per cent.

N. M. WALLER

**2115. A laboratory distillation method for the evaluation of crude glycerin.** F. A. Schlachter and H. D. Hoffman (*J. Amer. Oil Chem. Soc.*, 1954, **31** [5], 174-175).—The method described allows an evaluation of the yield of glycerol from a crude sample and also observation of the distillation behaviour of the material under simulated plant conditions. A 150-g sample of crude glycerin adjusted to 0.20 per cent. of free alkali (as  $\text{Na}_2\text{O}$ ) is subjected to vacuum distillation,  $\text{H}_2\text{O}$  being removed at  $100^\circ\text{C}$  and a pressure of 15 mm of Hg; the glycerin is then distilled at 6 to 8 mm of Hg with bath temp. rising from  $175^\circ$  to  $220^\circ\text{C}$  at the end of distillation. The glycerol content of the distillate is determined by sp. gr. measurement.

N. M. WALLER

**2116. The determination of small quantities of acetaldehyde.** H. Böhme and O. Winkler (*Z. anal. Chem.*, 1954, **142** [1], 1-5).—The colour of the dinitrophenylhydrazones of acetaldehyde obtained by the method of Lappin and Clark (*Brit. Abstr. C.*, 1951, 289) is unsuitable for colorimetric determination owing to fading; further, the quantity of HCl is critical. The determination is best carried out as follows. To 1 ml of acid dinitrophenylhydrazine soln. (50 mg in 50 ml of aldehyde-free methanol containing 0.4 ml of 38 per cent. HCl), add the aq. acetaldehyde soln. (1 ml) and set aside for  $\frac{1}{2}$  hr. at room temp.; add 5 ml of pyridine - water mixture (4 + 1) and 1 ml of KOH soln. (10 per cent. in 80 per cent. v/v methanol). Measure the extinction at 476  $\text{m}\mu$  after exactly 10 min. against a blank prepared similarly. This method can be used to estimate as little as 2 mg of acetaldehyde per litre of ethanol.

P. S. STROSS

**2117. Determination of aldehydes in the presence of ketones, and procedure for acetals.** H. Siegel and F. T. Weiss (*Anal. Chem.*, 1954, **26** [5], 917-919).—The method involves the reaction of the aldehyde with  $\text{Ag}_2\text{O}$ , formed *in situ* by the addition of NaOH to the aq. or alcoholic reaction mixture containing dil.  $\text{AgNO}_3$ . After acidification to redissolve the  $\text{Ag}_2\text{O}$ , the mixture is filtered and the unreacted  $\text{Ag}^+$  is determined by the Volhard method. Acetals are determined by the same technique, after hydrolysis with  $\text{H}_2\text{SO}_4$ . The accuracy in the presence of ketones is generally  $\pm 3$  per cent.

G. P. COOK

**2118. Gasometric determination of aldehydes and ketones.** A. P. Terent'ev and K. S. Zabrodina (*Compt. Rend. Acad. Sci., U.S.S.R.*, 1953, **92** [6], 1181-1184).—A method of determination of carbonyl groups in aldehydes and ketones is described. After the substance has reacted with phenylhydrazine in pyridine solution at  $80^\circ\text{C}$ , the excess of phenylhydrazine is decomposed by addition of  $\text{Cu}(\text{NO}_3)_2$ . The N evolved is collected and measured and the percentage of carbonyl group is estimated by comparison with the N collected in a blank test. To prevent decomposition of phenylhydrazones by the excess of  $\text{Cu}(\text{NO}_3)_2$ , some  $\text{FeSO}_4$  is added before heating the solution with ether to expel the N.

S. K. LACHOWICZ

**2119. Effect of side chain on the chromatographic adsorption of some ketones on carbon.** E. D. Smith and A. L. LeRosen (*Anal. Chem.*, 1954, **26** [5], 928-929).—A simple method for determining  $R_F$  values on coloured adsorbent materials is described. The method was applied to the study of side chain effect on the adsorption on C of some ketones. In

general the results show that the adsorption of the aliphatic straight-chain methyl ketones on C becomes progressively stronger as the length of the side chain is increased and it appears that this must be primarily due to the strong affinity of C for the organic side chains.

G. P. COOK

**2120. Methods of separating different fatty acids.** R. Rigamonti (*Olivi Min.*, 1954, **31** [4], 62-71).—The methods of preparing pure fatty acids are reviewed. The theoretical principles of fractional distillation, fractional crystallisation, chromatography, and selective-solvent extraction (including counter-current extraction) are discussed.

E. HAYES

**2121. Spray reagent for the detection of carbohydrates.** R. U. Lemieux and H. F. Bauer (*Anal. Chem.*, 1954, **26** [5], 920-921).—The spray reagent is prepared shortly before use by mixing 4 parts of 2 per cent. aq.  $\text{NaIO}_4$  and 1 part of 1 per cent.  $\text{KMnO}_4$  in 2 per cent. aq.  $\text{Na}_2\text{CO}_3$  soln. The presence of  $\text{IO}_4^-$  or  $\text{MnO}_4^-$ -reducing substances on paper strip chromatograms results in the formation of a greenish yellow spot after a short period of time. The reagent compares favourably in sensitivity with many of the reagents commonly used to detect reducing sugars.

G. P. COOK

**2122. Quantitative separation of oligogalacturonic acids by the use of ion exchangers.** R. Derungs and H. Deuel (*Helv. Chim. Acta*, 1954, **37** [3], 657-659).—A mixture of mono-, di-, tri- and tetragalacturonic acid is quant. separated as formates by means of the anion exchange resin Dowex 3 by elution with aq. solutions of formic acid of increasing concn.

H. WREN

**2123. On the paper chromatographic detection of fugitive aliphatic amines.** W. Dihlmann (*Biochem. Z.*, 1954, **325** [4], 295-298).—A method for the chromatographic analysis of saturated and unsaturated amines is reported. Ascending chromatograms are run with *n*-butanol - acetic acid - water (4:1:5). The amines were used in the form of the hydrochlorides (1 per cent. soln.). Spots were developed with ninhydrin soln. (0.2 per cent. in butanol) after drying at  $80^\circ$  to  $100^\circ\text{C}$ , or with Na 2-naphthaquinonesulphonate (1 per cent.) in NaOH (5 per cent.) after drying at room temp. Tertiary amines were detected by iodine vapour. The use of Na 2-naphthaquinonesulphonate permitted the distinction of saturated and unsaturated amines. Details are given of the colour reactions and  $R_F$  values of a number of amines.

G. W. CAMBRIDGE

**2124. Determination of organic sulphides by potentiometric titration.** V. G. Luk'yanitsa and A. S. Nakrasov (*Compt. Rend. Acad. Sci. U.S.S.R.*, 1953, **90** [6], 1043-44).— $\text{KIO}_3$  reacts with aliphatic sulphides thus:  $\text{KIO}_3 + 2\text{R}_2\text{S} + 2\text{HCl} = 2\text{R}_2\text{SO} + \text{KCl} + \text{ICl} + \text{H}_2\text{O}$ , and is used in 90 per cent. acetic acid to titrate aliphatic sulphides potentiometrically when dissolved in a mixture of dibutyl phthalate (35 pt.), acetic acid (60 pt.), and water (5 pt.), containing  $\text{ICl}$  (0.005 N) and  $\text{HCl}$  (0.4 N); the error is  $\pm 1$  per cent.

R. C. MURRAY

**2125. Additivity of refraction dispersion and comparative evaluation of dispersiometric methods of determining aromatic hydrocarbons.** B. V. Ioffe (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 60-64).—A detailed criticism of the paper by Kazansky *et al.* (*Anal. Abstr.*, 1954, **1**, 1286).

G. S. SMITH

**2126. Cerium [ceric] sulphate oxidation of phenols.** W. R. Spencer and F. R. Duke (*Anal. Chem.*, 1954, **26** [5], 919-920).—The method is based on the ppt. formed when  $\text{Ce}(\text{SO}_4)_2$  reacts with phenols. An aq. soln. of between 25 and 100 mg of phenol is acidified with  $\text{H}_2\text{SO}_4$  to  $\approx$  pH 1. An excess of standard  $\text{Ce}(\text{SO}_4)_2$  is added and the excess is titrated rapidly with standard  $\text{FeSO}_4$  after 1 to 3 min. to a potentiometric end-point. Alternatively, the ppt. is filtered, washed with water, dried in vacuum at  $50^\circ\text{C}$  and weighed. Empirical factors are used in the conversion calculations, those of 12 common phenols being listed. The accuracy for phenol and *o*- and *p*-cresols is good.

G. P. COOK

**2127. Separation and identification of 2:4-dinitrophenylhydrazones by paper chromatography.** H. S. Burton (*Chem. & Ind.*, 1954, [20], 576).—Good separations of the 2:4-dinitrophenylhydrazones of widely differing carbonyl compounds are obtained by the use of paper chromatography. Portions of 1 to 5  $\mu\text{l}$  of a 1 in 1000 solution of the hydrazone in  $\text{CHCl}_3$ , methanol or ethanol are used, by the descending technique, with a 50 to 75 per cent. aq. soln. of ethyl lactate as the stationary phase, and development with 1 to 3 per cent.  $\text{CCl}_4$  in light petroleum. Separation occurs after 3 to 6 hr.

G. R. WHALLEY

**2128. Functional organic analysis. I. Thermo-gravimetric analysis of 2:4-dinitrophenylhydrazones.** C. Duval and N. D. Xuong (*Anal. Chim. Acta*, 1954, **10** [6], 520-522).—The 2:4-dinitrophenylhydrazones of 14 aldehydes and 19 ketones have been examined for thermal stability by heating to their m.p. on the thermobalance. With few exceptions and in the temp. ranges used, the compounds are sufficiently stable for gravimetric purposes.

W. C. JOHNSON

**2129. The micro-determination of picric acid in picrates.** P. R. W. Baker (*Analyst*, 1954, **79**, 289-292).—Three micro-scale methods for determination of picric acid in organic picrates have been examined. Bolliger's method (*Brit. Abstr. A II*, 1939, 398) of titration with methylene blue was found not to be of universal application. Titration with standard NaOH with ethyl bis-2:4-dinitrophenylacetate as indicator is rapid and simple, but applicable only to picrates known to be derived from non-basic substances. The macro method of Busch *et al.* (*Z. angew. Chem.*, 1908, **21**, 354) in which the picrate is pptd. as nitron picrate, collected and weighed gave satisfactory results on a micro scale and was applicable to all the compounds examined. The solubility of the ppt. and the effect of presence of Cl on its purity were examined. A. O. JONES

**2130. The determination of piperazine. VIII.** A. Castiglioni and M. Vietti (*Z. anal. Chem.*, 1954, **142** [1], 18).—The compound formed when piperazine is quant. pptd. with an excess of ammonium molybdate is shown to be  $3\text{C}_4\text{H}_{10}\text{N}_4 \cdot 10\text{MoO}_3 \cdot 8\text{H}_2\text{O}$ . To an aqueous piperazine soln. (5 ml), add glacial acetic acid (5 ml) and 5 per cent. ammonium molybdate (10 ml). Heat on a water-bath for 2 hr., set aside for 8 hr., filter and dry the ppt. at  $100^\circ\text{C}$ .

P. S. STROSS

**2131. Determination of 5-(hydroxymethyl)-2-furaldehyde and related compounds.** J. H. Turner, P. A. Rebers, P. L. Barrick and R. H. Cotton (*Anal. Chem.*, 1954, **26** [5], 898-901).—A study of the u.v. absorption characteristics of 5-(hydroxymethyl)-2-furfural and related compounds is given.

G. P. COOK

**2132. Infra-red absorption bands characteristic of the oxirane ring.** W. A. Patterson (*Anal. Chem.*, 1954, **26** [5], 823-835).—Infra-red absorption spectra of 26 oxiranes in the liquid state and in soln. have been recorded at 2 to 15  $\mu$ . The compounds examined included epoxy hydrocarbons, esters and ethers, as well as cyclic oxides and a number of dioxides. The characteristic strong band at  $\approx 8\ \mu$  ( $1250\text{ cm}^{-1}$ ) is confirmed and evidence is found of two other strong bands, also characteristic of the epoxide ring, at  $\approx 11$  and  $12\ \mu$ . The precise position of each of these three bands varies with the specific epoxy compound. In general, their positions can be correlated with the reactivities of the oxirane compounds with acetic acid at  $25^\circ\text{C}$ .

W. J. BAKER

**2133. Analysis of binary solvent mixtures of conducting solutions by a radio-frequency method.** J. L. Hall, J. A. Gibson, jun., F. E. Critchfield, H. O. Phillips and C. B. Seibert (*Anal. Chem.*, 1954, **26** [5], 835-838).—The composition of binary solvent mixtures containing high concn. of electrolyte is determined indirectly from measurements of the high-frequency (20 Mc) capacitance of the insulated cell and the specific conductance ( $\kappa$ ) and dielectric const. ( $\epsilon$ ) of the soln. inside the cell. For the system dioxan - water - KCl, with  $\kappa = 0$  to  $180 \times 10^{-5}$ , measured values of  $\epsilon$  are accurate to  $\pm 0.3$  and can be related directly to the composition of the binary mixture, so that empirical calibration curves for the system can be constructed. The method can be extended to other systems, e.g., methanol, tetrahydrofuran, acetone or *tert*-butanol - water - KCl, by using calibration curves based on one system. Factors affecting the accuracy of the results (which is less when the solvent  $\epsilon$  is  $< 50$ ) are discussed.

W. J. BAKER

**2134. Determination of alkylchlorosilanes in the air.** E. A. Peregrin and N. P. Kozlova (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 47-50).—Optimum conditions for the use of ascorbic acid in the molybdenum-blue method for determining Si are studied. For determining concn. of vapours of alkylchlorosilanes in air, the air is passed through  $\text{H}_2\text{SO}_4$  absorbents, the solution is evaporated, the residue is fused with  $\text{Na}_2\text{CO}_3$  -  $\text{K}_2\text{CO}_3$ , the melt is dissolved in dil.  $\text{H}_2\text{SO}_4$ , and the solution is treated with ammonium molybdate, tartaric acid and ascorbic acid.

G. S. SMITH

**2135. Proposed method of test for water in petroleum and bituminous products.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 358-364).—The Dean and Stark method is recommended. When a glass still is used, 5 per cent. v/v of the solvent must distil at  $194^\circ$  to  $212^\circ\text{F}$  and 90 per cent. at  $< 410^\circ\text{F}$ ; with a metal still a solvent of which 98 per cent. v/v distils at  $248^\circ$  to  $482^\circ\text{F}$  may be used. For tarry or asphaltic materials a mixture of benzene ( $\approx 20$  per cent.) and xylene ( $\approx 80$  per cent.) may be used. The reproducibility should be within 0.2 per cent. for petroleum products or asphalt emulsions containing 1 to 25 per cent. of water and within 0.4 per cent. for asphalt emulsions with water contents of 25 to 50 per cent.

J. M. JACOBS

**2136. Proposed method of test for hydrocarbon types in jet propulsion fuels. Fluorescent-indicator adsorption (FIA) method.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 353-357).—A procedure is described for determining aromatics, olefines and saturates in

aircraft gas turbine and jet propulsion fuels having distillation end-points  $> 600^{\circ}\text{F}$ . Compounds containing S, N or O are determined as aromatics. The sample ( $\approx 0.75\text{ ml}$ ) containing traces ( $\approx 1$  in 1000 by vol.) of an added fluorescent dye mixture (Sudan III, together with an olefine and aromatic dye dissolved in xylene) is introduced into a narrow glass column packed with activated silica gel. The sample is then forced down the column by adding isopropanol and applying a gas (air or N) pressure of 15 lb per sq. in. The boundary of each hydrocarbon type is observed in u.v. light. Duplicate test results should not differ by  $> 1.5$  per cent. and reproducibility (as determined by average results obtained in two laboratories) should be within 2.5 per cent.

J. M. JACOBS

**2137. Proposed methods of test for API gravity and specific gravity of petroleum and its products by hydrometer.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 347-349; 350-352).—A procedure is described in detail for the determination, by means of a glass hydrometer, of the API gravity (*in vacuo*) or the sp. gr. (*in vacuo*) of crude petroleum and liquid petroleum products having a Reid v.p. of  $> 26\text{ lb}$ . The test is carried out at  $60^{\circ}\text{F}$ , or the results determined at any convenient temp. from  $0^{\circ}$  to  $195^{\circ}\text{F}$  are converted to values at  $60^{\circ}\text{F}$  by means of standard tables.

J. M. JACOBS

**2138. Use of fractionation methods for the analysis of petroleum products.** A. Crozier (*Rev. Inst. Franç. Pétrole*, 1954, **9** [2], 42-50).—The bibliography of various physical methods including distillation (with various types of column), selective adsorption, extractive crystallisation with urea, thermal diffusion, etc., for the fractionation of petroleum products is reviewed (108 references).

J. M. JACOBS

**2139. Proposed method for polarographic determination of tetraethyl-lead in petrol.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 365-369).—The sample (25 to 100 ml, diluted with heavy distillate if the  $\text{Pb}(\text{C}_2\text{H}_5)_4$  content is  $> 2\text{ ml}$  per U.S. gall.) is refluxed with 50 ml of conc. HCl for 30 min. to convert the Pb to  $\text{PbCl}_2$ . Known quantities of  $\text{CdCl}_2$  and of gelatin are added to the acid extract and the Pb content of an aliquot is determined polarographically by use of a silver anode.

J. M. JACOBS

**2140. Proposed manometric method with null pressure transmitter for vapour pressure test of petroleum products.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 377-379).—This method is proposed as an addition to A.S.T.M. Method D 323 (Reid method), the null-pressure transmitter (two gas chambers separated by a flexible impervious membrane) being substituted for the gauge. A contact indicator, consisting of 3 neon lamps, each in series with a 200,000-ohm resistor, is connected to the transmitter and the vent valve is closed. The air valve is then opened, to apply a pressure nearly balancing that in the Reid bomb, to obtain an approx. reading on the manometer. The final adjustment of the null point is obtained by opening the vent valve slightly until a state of static balance is indicated by the blinking of the neon lamp. The pressure on the mercury manometer at this point gives a figure for the Reid v.p. within the bomb.

J. M. JACOBS

**2141. Cryoscopic method of determination of total amount of arenes and unsaturated hydrocarbons in**

**kerosene and gasoline fractions.** M. D. Tilicheev and N. A. Okinshevich (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 3-10).—Unsaturated hydrocarbons in hydrocarbon mixtures treated with  $\text{H}_2\text{SO}_4$  tend to form polymers that remain in the hydrocarbon layer, but they are completely removed together with the aromatic hydrocarbons when  $\text{P}_2\text{O}_5$  in  $\text{H}_2\text{SO}_4$  (30 g of  $\text{P}_2\text{O}_5$  in 100 ml of 100 per cent.  $\text{H}_2\text{SO}_4$ ) is used for the extraction. The method is applied to the determination of total arenes and alkenes in petroleum fractions within the boiling range  $150^{\circ}$  to  $400^{\circ}\text{C}$  by the cryoscopic technique described previously (*Brit. Abstr. C*, 1953, 160).

G. S. SMITH

**2142. Determination of total sulphur by reductive decomposition.** W. Radmacher and P. Mohrhauser (*Z. anal. Chem.*, 1954, **141** [6], 419-429).—Sulphur can be assayed reliably in fuel oils, tar oils and many solid fuels by reduction to sulphide with Li; this is best done in a special bomb. The sulphide is oxidised quant. to sulphate with dichromate. As and P do not interfere. Heat to  $1000^{\circ}\text{C}$  for 15 min. a quantity of material containing about 16 mg of S in a bomb with 800 mg of Li. Transfer the mass to a distillation flask, add phosphoric acid,  $\text{CrCl}_3$  or  $\text{TiCl}_3$ , and distil into NaOH, which is allowed to become hot. Run the alkaline sulphide soln. slowly into excess of standard  $\text{K}_2\text{Cr}_2\text{O}_7$ , maintaining the temp. between  $70^{\circ}$  and  $90^{\circ}\text{C}$  and titrate the excess of dichromate with ferrous sulphate. The end-point is determined potentiometrically. An iodimetric method of determining the sulphide can also be used.

P. S. STROSS

**2143. Quantitative determination of sulphur in organic substances and fuel in presence of chromium oxide catalyst.** P. N. Fedoseev and R. M. Lagoshnaya (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 37-41).—The use of  $\text{Cr}_2\text{O}_3$  ensures complete oxidation to  $\text{SO}_2$  of S in organic substances burnt in O. The products of combustion are collected in a previously evacuated vessel containing a known vol. of 0.1 N alkali and 2 to 3 ml of 3 per cent.  $\text{H}_2\text{O}_2$ .

G. S. SMITH

**2144. Analysis of gas oil and cycle stock from catalytic cracking.** E. M. Charlet, K. P. Lanneau and F. B. Johnson (*Anal. Chem.*, 1954, **26** [5], 861-871).—The oil was first separated into aromatic and non-aromatic portions by adsorption on silica gel and use of *n*-heptane for the non-aromatic elution and acetone for the aromatic. The two separate fractions were stripped of solvent and the aromatic portion was separated into 9 fractions by distillation at absolute pressures of 0.08 to 0.15 mm of Hg, and each of these fractions was then separated further by desorption from an alumina column with acetone as solvent, which was subsequently stripped from the fraction. The fractions were examined by u.v. analysis, and these results in conjunction with i.r. absorption, molecular wt. and C-H determinations enabled a quant. analysis to be carried out. The compositions are presented in terms of wt. per cent. of each molecular type found in the aromatic portion of the oils, and they are classified on the basis of the configuration of the aromatic rings in the molecules. Eleven u.v. spectra are illustrated and tables giving the typical aromatic hydrocarbons found in these oils are also included.

G. P. COOK

**2145. Proposed method of test for tensile strength of paraffin wax.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 371-376).—An empirical measure of the tensile

strength (longitudinal stress required to break a specimen of specified shape and cross-section) of paraffin wax having a ductility  $\geq 0.125$ -in. elongation at the temp. and R.H. specified for the test and having A.S.T.M. m.p. of  $120^\circ$  to  $150^\circ$  F (A.S.T.M. method D87) is described. A set of 6 specimens, cast by means of a mould of specified size and shape, are aged for 2 hr. at  $73^\circ$  F and 50 per cent. R.H., and a load of 20 lb per sq. in. is applied by a testing machine, which is accurate to 1 per cent. for the lowest load to be applied. The manner and speed of testing the shape and cross-section of the specimen, and the manner of prep., conditioning, and surface condition of the specimen all influence the magnitude and precision of the test results. Within the range 150 to 450 lb per sq. in., the repeat tests (mean of a series of 6) should not differ by  $> 24$  lb per sq. in. and should be reproducible to within 36 lb per sq. in.

J. M. JACOBS

**2146. Proposed method of test for oil content of petroleum waxes.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 380-383).—The test is intended for the determination of oil in petroleum waxes having a m.p. of  $< 105^\circ$  F, and an oil content of  $\geq 15$  per cent. The sample is warmed with ethyl methyl ketone and stirred with a wire stirrer until solution is complete or (for high-melting microcrystalline waxes) until the undissolved material is well dispersed as a fine cloud. The wax-solvent slurry is cooled to  $-25^\circ \pm 0.5^\circ$  F, to precipitate the wax, and is then filtered through a sintered-glass filter stick. The solvent is evaporated from the filtrate at  $95^\circ \pm 2^\circ$  F until the loss between successive weighings is  $\geq 0.2$  mg.

J. M. JACOBS

**2147. Interrelation and application of laboratory and works data [on coal processing]. H. Crude benzole in laboratory and refinery. [Determination of cyclopentadiene in first runnings of benzole.]** W. Mantel and H. Hansen (*Brennstoff-Chem.*, 1954, **35** [7-8], 97-104).—To 20 ml of 0.1 N maleic anhydride in toluene in a stoppered flask is added 5 ml of pre-dried ( $\text{CaCl}_2$ ) distillate. After 1 hr. with frequent shaking for completion of the condensation, 15 ml each of aq. KI (4 per cent.) and  $\text{KIO}_3$  (24 per cent.) and 50 ml of 0.1 N  $\text{Na}_2\text{S}_2\text{O}_3$  are added. After 2 hr. more, the residual thiosulphate is titrated with 0.1 N I to a brown colour and back-titrated with 0.1 N thiosulphate. A blank test is done. If  $x$  ml of 0.1 N thiosulphate correspond with  $y$  ml of distillate, the content of cyclopentadiene is  $0.33 x/y$  g per 100 ml.

A. R. PEARSON

**2148. Determination of solid and liquid impurities in synthesis gas.** L. J. Kane, H. W. Wainwright, C. C. Shale and A. E. Sands (*U.S. Bur. Min. Rep. Invest.*, 1954, No. 5045, 23 pp).—Methods of determining solid and liquid impurities in synthesis gas produced from coal are described. With crude gas, correct sampling is difficult, and methods of checking the sampling technique and preventing blockages in the sampling tubes are described. Types of filter-paper for high and low dust concn., shaping of filters and conditioning for accurate weighing are discussed. With wet gas some of the dust is first condensed with the water, and is determined after evaporation and washing. Microscopic particle size analysis is discussed. Low dust concn. may be determined by a photometric method without sampling.

A. B. DENSHAM

**2149. A semi-micro method for determination of formaldehyde viscose or cuprammonium rayon treated with formaldehyde, or with urea- or melamine-formaldehyde.** Anon. (*Shirley Inst. Test Leaflet, Chem.* 20, First Edition, March, 1954).—A measured vol. of 12 N  $\text{H}_2\text{SO}_4$  is added to a weighed sample of rayon, and the soln. is filtered or decanted after at least 4 hr. A 50-ml aliquot of the filtrate is treated with 1 ml of 5 per cent. chromotropic acid and 5 ml of conc.  $\text{H}_2\text{SO}_4$  in a boiling tube standing in boiling water for 20 to 30 min., cooled and again made up to 50 ml with distilled  $\text{H}_2\text{O}$ . The optical density of the red-violet solution is measured on an absorptiometer, and the amount of formaldehyde corresponding to this result is obtained from a curve previously determined from standard formaldehyde solutions. A. M. SPRATT

**2150. A colour test for Nylon 66 and for Terylene.** W. J. Roff (*Analyst*, 1954, **79**, 306-307).—The colour test described provides a distinction between Nylon 66 (polyhexamethylene adipamide) and Terylene (polyethylene terephthalate) and differentiates them from other commercial fibres. It depends upon a reaction between alkaline o-nitrobenzaldehyde and the pyrolytic vapours from the test materials. Positive results are given also by any polyamide containing adipic acid residues, by adipic acid itself and certain of its derivatives and by non-fibrous Terylene and related polyesters derived from ethylene glycol and from polyvinyl alcohol. A few mg of the sample are gently heated in a vertically clamped test tube until heavy vapours rise to the upper part of the tube ( $350^\circ$  to  $400^\circ$  C). A strip of filter-paper moistened with a saturated soln. of the reagent in 2 N NaOH is then brought into contact with the vapour. Nylon 66 gives a deep mauve-black colour discharged by dil.  $\text{H}_2\text{SO}_4$ ; Terylene gives a greenish blue colour changing to blue or pale indigo with dil.  $\text{H}_2\text{SO}_4$ . The colour with Nylon 66 appears to be due to presence of cyclopentanone in the vapour; with Terylene the blue compound appears to be indigo.

A. O. JONES

**2151. Analysis of textile auxiliary products.** J. A. van der Hoeve (*J. Soc. Dyers Col.*, 1954, **70**, 145-154).—A new classification (36 groups) and an analytical scheme for the analysis of textile auxiliary products is described. After inorganic salts have been separated, organically bound N is determined, the ionic nature of the compound (cation-active, anion-active, non-ionic) is found and the hydrocarbon type is determined. Many of the conventional tests have been modified and some new ones added; a scheme for the analysis of mixtures is included.

E. S. LANE

**2152. Proposed methods of test for surface and interfacial tension of solutions of surface-active agents.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 447-451).—Measurements are made with a du Nouy precision tensiometer or interfacial tensiometer equipped with a 4 or a 6-cm platinum ring. Directions for the calibration of the apparatus are given.

J. M. JACOBS

**2153. Determination of alcohols [used] in perfumery. Practical method for the evaluation of tertiary alcohols.** H. Ott (*Helv. Chim. Acta*, 1954, **37** [3], 786-787).—The alcohol (e.g., linalol) (5 ml), pure pyridine (10 ml) and 98 per cent. acetic

anhydride (30 ml) are heated to gentle boiling in 15 to 20 min. and kept at this temp. for 5 hr. The mixture is then cooled, shaken with water and then with 1 per cent. aq.  $\text{NaHCO}_3$ . The ester is dried and the saponification no. is determined. When compared with the results obtained by the cold formylation method, the figures obtained are somewhat low, but they are consistent, so a mathematical correction can be applied. H. WREN

**2154. The oil content of tung products by a rapid petroleum-naphtha method.** R. S. McKinney and R. L. Holmes (*J. Amer. Oil Chem. Soc.*, 1954, **31** [5], 172-174).—The method described gives results in good agreement with the 4-hr. extraction procedure with light petroleum, and has the added advantage that the solvent has a high flash-point (100° F). A 50-g finely ground sample (of tung fruit, press-cake or filter-cake) is mixed mechanically with 100 g of high-boiling petroleum naphtha (sp. gr. 0.7817 at 25° C; boiling range 312° to 387° F) at 25° C for 10 min. The soln. of the oil is filtered and its sp. gr. is determined by means of a pycnometer or sp. gr. balance. The oil content is determined by reference to standard tables or graphs. N. M. WALLER

**2155. Rosin acids. I. Titrimetric determination with bromine in methanol of rosin acids with two double bonds in pure rosin acid mixtures.** P. O. Jalava (*Finnish Paper & Timber J.*, 1954, **36**, 69-70).—One g of mixed rosin acids is titrated with 0.2 N Br in methanol, the end-point being indicated by the yellow colour of unreacted Br. The rosin acids with two double bonds react instantaneously with Br. Dehydro- and tetrahydroabietic acids do not react at all and dihydroabietic acid reacts only slowly. S. V. SERGEANT

**2156. Electrical measurements in the study of immersed paint coatings on metal. I. Comparison between capacitance and gravimetric methods of estimating water-uptake.** D. M. Brasher and A. H. Kingsbury (*J. Appl. Chem.*, 1954, **4** [2], 62-72).—Parallel tests are made to estimate the water uptake of 12 different paints on panels of nickel foil immersed in sea-water. In one series of tests the percentage uptake of  $\text{H}_2\text{O}$  is calculated from capacitance measurements by application of the formula of Hartshorn *et al.* (*J. Soc. Chem., Ind.*, 1937, **56**, 266r). These values are compared with those found gravimetrically in the other series of tests. Usually agreement between the results is good, but occasionally there is a pronounced disagreement. No clear relationship appears to exist between the amount of agreement and the type of paint formulation. Differences between the results sometimes suggest the absence of the completely random distribution of water within the paint film that is assumed in the calculation from capacitance values. The capacitance method of measuring water uptake should prove useful for routine testing on paints made to a formulation that is known to give good agreement between the two methods. It has the advantages of speed and simplicity, and does not require the many specimens that the gravimetric method does. J. H. WATON

**2157. Nutrition of *Hevea Brasiliensis*. I. Experimental methods.** E. W. Boile-Jones (*J. Rubber Res. Inst. Malaya*, 1954, **14**, 183-207).—The estimation of chlorophyll in the laminae of *Hevea*

*Brasiliensis*—The transmittance of a chlorophyll solution at 642.5 and 660  $\text{m}\mu$  was determined, and the concentration of chlorophyll was calculated. This result was used in preparing a calibration curve for the colorimetric estimation of chlorophyll.

*The estimation of rubber in petiolar tissues*—A rapid method is described for the routine estimation of rubber in petiole samples. Dried petiole is extracted with benzene (boiling range 50° to 60° C), and the filtered extract is evaporated to dryness. The residue is refluxed with alcoholic NaOH, the supernatant liquid is then decanted and a formic acid solution is added to the rubber film. The rubber is filtered off, washed with water and ethanol, and dried to constant weight.

*The determination of sugars in laminae of Hevea Brasiliensis*—A chromatographic method was used to estimate each sugar present in alcoholic leaf extracts. Chromatograms were prepared by use of a butanol-ethanol-water mixture as solvent, and then dipping the papers in  $\text{AgNO}_3$  solution. Separation was sufficient to distinguish glucose, fructose, sucrose and inositol. When the papers were dried, the spots were delineated, cut out, oven-dried and weighed. By reference to a standard curve prepared under similar conditions the amount of sugar present was determined. It was necessary to prepare calibration curves for each batch of estimations. D. LIFF

**2158. Determination of particle-size distribution in GR-S latexes.** A. Nisonoff, W. E. Messer and L. H. Howland (*Anal. Chem.*, 1954, **26** [5], 856-861).—The wt. distribution of particles of diam. 50 to 2000  $\text{m}\mu$  in rubber latex is determined, with a probable error of  $\pm 2.5$  per cent., by using an International Type SB-1 centrifuge and applying Stokes' law. The latex is diluted to contain 2 per cent. of solids and is centrifuged at 2700 r.p.m. in a flat-bottomed tube (50 ml), the temp. being controlled to  $\pm 2^\circ \text{C}$  of room temp. during centrifugation. The viscosity and  $d_r$  are determined separately. By varying the time of centrifuging, the centrifugal force, and the height from which the sample is removed for determination of solids concn., results are obtained from which the integral wt. distribution curve can be constructed. Particles of diam.  $> 2000 \text{ m}\mu$  are investigated by reducing the time or speed of centrifugation, or by using gravitational settling. Curves for several samples of synthetic rubber latex are shown and discussed briefly. W. J. BAKER

**2159. A comparison of methods of determining the percentage basicity of one-bath chrome liquors.** D. Burton and M. C. Thompson (*J. Soc. Leath. Tr. Chem.*, 1954, **38** [3], 102-105).—Details are given of the Procter-McCandlish, the Burton, Glover and Wood, and the Lehigh (Thorstensen and Theis) methods of determining the percentage basicity of one-bath chrome liquors. Theoretical and observed results from all three methods are tabulated. An erroneously high value is given by the Procter-McCandlish method where anions have entered the Cr complex owing to pptn. of basic Cr salts. The Burton, Glover and Wood method gives satisfactory results with "straight" chrome liquors, but is not applicable to liquors containing masking salts oxidisable by  $\text{H}_2\text{O}_2$  to  $\text{CO}_2$ . The Lehigh method gives satisfactory results with all soln., but for accurate results a finely calibrated burette and a pH meter are necessary. O. M. WHITTON

## 4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD,  
SANITATION, AGRICULTURE

## Blood, Bile, Urine, etc.

**2160. Method for determination of total amount of haemoglobin and blood volume in small animals.** C. A. Gemzell and T. Sjöstrand (*Acta Physiol. Scand.*, 1954, **30** [4], 369-374).—A method for the estimation of the total amount of haemoglobin in small animals is described. The method is based on the determination of the absorption of small amounts of CO by the animal during certain standard conditions. Blood is not withdrawn from the animal and the estimations could be repeated every second or third day throughout a period of several months. If the haemoglobin concentration of the blood is simultaneously determined, the blood volume may also be estimated. In a single determination the method has an error of  $\pm 6$  per cent. as judged by duplicate determinations. The application of the method and its reliability are demonstrated by experiments with bleeding and by following the values of total haemoglobin during body growth of 16 rats. The total haemoglobin shows a linear correlation with the body weight up to 250 g.

I. JONES

**2161. Paper chromatography of free amino-acids in blood plasma.** S. Gordon and G. L. Nardi (*J. Lab. Clin. Med.*, 1954, **43** [5], 827-830).—A method for preparing blood plasma or serum for two-dimensional paper chromatography is given. The free  $\alpha$ -amino-acids and ninhydrin-reacting substances found in normal human blood by this method are described.

W. H. C. SHAW

**2162. The estimation of serum proteins by electrophoresis on filter-paper.** J. Hardwicke (*Biochem. J.*, 1954, **57** [1], 166-171).—A method is described for quant. electrophoresis of proteins on filter-paper. It is a modification of the method of Turba *et al.* (*Naturwiss.*, 1950, **37**, 93). The following variables inherent in the method are examined critically: (i) staining and washing of the papers (0.1 per cent. of bromophenol blue is used for staining; washing with 0.5 per cent. acetic acid gives max. dye density per unit concn. of protein); (ii) reproducibility of staining and optimum concn. of protein solutions; (iii) "tailing" of proteins on the paper during migration; and (iv) dye-binding capacity of different protein fractions. The accuracy of the method is  $\pm 6$  per cent. of the total protein present. The filter-paper method, compared with the classical Tiselius method, is much simpler and more rapid, and it may be more accurate in the analysis of pathological sera that contain high proportions of lipid or carbohydrate.

J. N. ASHLEY

**2163. A spectrometric method for the differential determination of serum proteins.** M. N. Mikhail and M. K. Salah (*Acta Pharm. Int.*, 1953, **2** [5], 419-423).—Serum on treatment with half-saturated  $(\text{NH}_4)_2\text{SO}_4$  yields a ppt. of globulin, which on washing and drying followed by solution in 0.1 N NaOH shows an absorption band at 290  $\text{m}\mu$  with  $E_{1\%}^{1\text{cm}}$  = 16.8. Isolation of the albumin by alcohol pptn. and washing followed by solution in half saturated  $(\text{NH}_4)_2\text{SO}_4$  shows at 277.5  $\text{m}\mu$  an  $E_{1\%}^{1\text{cm}}$  of 8.6. In the proposed assay, 0.1 ml of serum is

treated in a centrifuge tube with 10 ml of half-saturated aq.  $(\text{NH}_4)_2\text{SO}_4$ , shaken, set aside (10 min.) and centrifuged. The clear liquor is carefully decanted, shaken with ether (to extract cholesterol and carotenoids) and separated, and the spectrophotometric reading is determined; 11.6 times this value gives the percentage w/v of serum albumin. The solid globulins in the centrifuge tube are dissolved in 10 ml of 0.1 N NaOH, and the spectrophotometric reading is multiplied by 6 to give the percentage w/v of serum globulins. The factors used are calculated from the absorption figures.

F. R. MUMFORD

**2164. Investigation of the paper-chromatography of human serum proteins.** F. Kazmeier and A. Gassen (*Klin. Wochschr.*, 1954, **32** [3-4], 81-85).—Two-dimensional paper chromatography of normal serum has not given reproducible results, but unidimensional chromatography with or without the addition of surface-active substances is satisfactory. Serum (0.02 ml) diluted with an equal volume of dist.  $\text{H}_2\text{O}$  with or without 1, 2 or 10 per cent. of a surface-active agent such as Tween has been used on a 20-cm strip of Whatman No. 1 paper. The following solutions have been used: Na barbitone buffer (pH 8.6), half-saturated  $(\text{NH}_4)_2\text{SO}_4$ , Ringer's soln., 0.1 M Na K tartrate, 0.1 M fructose or a combination of these, but in the main 0.1 M cane sugar solution has been found to give good separation of the components. Bromothymol blue has been used to indicate the proteins. Normal serum shows a characteristic distribution, slightly modified by the various surface-active agents, and deviations from the normal can be detected although the components of the various zones have not been identified.

G. W. CAMBRIDGE

**2165. Assay of plasma insulin activity by the rat-diaphragm method.** P. J. Randle (*Brit. Med. J.*, 1954, **1**, 1237-1240).—The presence of insulin in human plasma can be detected and assayed by its potentiating action on the uptake of glucose by the isolated normal rat diaphragm *in vitro*. For a four-point assay, the absolute uptake of the rat hemidiaphragms in the presence of 2 dilutions of plasma is compared with 2 standard doses of insulin. The hemidiaphragms are incubated in shaking manometer flasks for 3 hr. at 37°C in the presence of a buffer glucose mixture. The residual glucose is then determined in the substrate from which the uptake of glucose as mg of glucose per g weight of wet diaphragm per hour is calculated. The minimum sensitivity to insulin is 0.1 milli-unit per ml. The slope  $b$  of the log-dose response curve is  $0.6925 \pm 0.033$ , the standard deviation  $s$  of the points about the regression line is  $\pm 0.25$  and the index of precision  $\lambda = (s/b)$  is 0.36. The method is not therefore very precise. Results are reported for the insulin activity of normal human plasma, which was 13 milli-units per ml after a glucose meal. Evidence is presented of the presence in human plasma of a material having an insulin-like activity on the rat diaphragm.

G. F. SOMERS

**2166. Estimation of insulin levels in blood-plasma.** C. Goldi (*Schweiz. med. Wochschr.*, 1954, **84** [9], 276).—The method of estimating insulin in blood plasma by its effect on the glucose uptake of the rat diaphragm *in vitro* (Vallance-Owen and Hurlock, *Lancet*, 1954, **i**, 68) has been investigated.

The glucose consumption of the diaphragm in any one experiment is approx. constant (variation is  $\pm 3.5$  per cent.), but the basal value varies as much as 30 per cent., hence the response to standard doses of insulin must be checked each day. The relation between glucose uptake and insulin dosage is linear provided that 5-times crystallised insulin is used, the curve being irregular if commercial insulin preparations are used. Normal fasting plasma insulin levels were 40 to 80 milli-units per ml.

G. W. CAMBRIDGE

**2167. Further investigations on the lipopeptides of blood.** W. Schrader, G. Becker and E. Böhle (*Klin. Wochschr.*, 1954, **32** [1-2], 27-33).—Lipopeptides extracted from serum have been separated by paper chromatography and analysed for their component amino-acids. The best separation was achieved by two-dimensional chromatography: 10 to 12 hr. in butanol, acetic acid and water (4:1:5), and 5 to 8 hr. in methanol, acetic acid and water (98:1:1). The spots were developed by ninhydrin, 9 to 11 separate peptides being obtained.

G. W. CAMBRIDGE

**2168. Acidosis-producing metabolic disturbances and blood alcohol assays after brain-injury.** P. Seifert, R. Lambrecht and H. Manck (*Disch. med. Wochschr.*, 1954, **79** [5], 193-195).—Blood-alcohol determinations by Widmark's method (distillation of the alcohol and its oxidation by dichromate- $H_2SO_4$ ) is known to be subject to errors if keto-acids are present. It was thus of interest to know if acidosis occurring after head injury influenced blood alcohol estimation. In 80 cases of head injury 10 per cent. showed acidosis. The blood alcohol values were normal during this period of acidosis and false positives were not obtained.

G. W. CAMBRIDGE

**2169. Rapid determination of salicylate in biological fluids.** P. Trinder (*Biochem. J.*, 1954, **57** [2], 301-303).—The method, which is applicable to cerebrospinal fluid, plasma, whole blood and urine, depends on the purple colour given by salicylic acid in presence of  $Fe^{III}$ . The reagent mixture of  $Fe(NO_3)_3$ ,  $HgCl_2$  and aq.  $HCl$  precipitates the proteins. After centrifuging, the optical density is determined on a photo-electric colorimeter with Ilford green 624 filter (or Ilford green 404, or Chance OG1 filter) or spectrophotometrically at 540  $m\mu$ , the amount of salicylic acid then being ascertained from a standard graph. Control tests on normal sera and plasma give colours equivalent to < 1.1 mg of salicylic acid per 100 ml. A single determination requires only 5 min. Presence of 100 mg of  $PO_4^{III}$ , 20 mg of bilirubin, 25 mg of phenol, 10,000 i.u. of heparin, or 1 g of glucose or urea, per 100 ml of serum does not interfere with the determination. A slightly high value is obtained in presence of oxalates or ethyl acetate. When a Spekker absorptiometer is used, 0.2 ml of blood is sufficient for a determination.

J. N. ASHLEY

**2170. A rapid method of identifying particular barbiturate derivatives in small samples of blood by paper chromatography.** J. T. Wright (*J. Clin. Pathol.*, 1954, **7** [1], 61-65).—Rectangular strips of Whatman No. 1 filter-paper 15  $\times$  50 cm are sprayed with Clark's borax buffer, pH 10.6, air-dried and suspended in a closed tank in an atmosphere saturated with  $H_2O$  and  $CHCl_3$  vapour for 1 hr. Barbituric acid derivatives (I) in 0.25 per cent. alcoholic soln. are applied to the base

line 2.5 cm apart, the paper is suspended in the tank with its upper end immersed in redistilled  $CHCl_3$ , and the chromatogram (descending) is developed for 4 to 7 hr. The paper is air-dried and the spots of I are detected by u.v. light. For chromatography of blood extracts, the acid soln. used for u.v. spectrophotometry is extracted with ether, and the residue from ether evaporation is dissolved in alcohol to give 0.25 per cent of I. Crude extracts obtained by direct extraction of blood with ether may also be used if the approx. concn. of I is known. Thirteen different specimens of I were found to be separable and identifiable by this method. The relative rates of travel of I were directly related to the relative speeds of their clinical action.

H. F. W. KIRKPATRICK

**2171. Photometric micro-determination of plasma fibrinogen with a thrombin-ninhydrin procedure.** A. Saifer and A. Newhouse (*J. Biol. Chem.*, 1954, **208** [1], 159-179).—A photometric micro-method is described for determination of plasma fibrinogen. It is based on Morrison's technique of clotting, syneresis and washing (*Brit. Abstr. C*, 1948, 143), and requires a ninhydrin procedure for the determination. The colour density is determined spectrophotometrically at 570  $m\mu$ . The method determines 0.3 to 2.0 mg of fibrinogen and the results are within  $\pm 5$  per cent. of those obtained by the macrogravimetric method. A new, partly hydrolysed fibrinogen standard is prepared from normal plasma. It remains stable indefinitely, and the fibrinogen content is standardised by the gravimetric or micro-Kjeldahl methods. By using aliquots of this standard, together with controls for each set of unknowns, an accurate colorimetric factor is determined for each analysis instead of the arbitrary factors used in the tyrosine-phenol reagent method. A method is described for determination of the occlusion error which permits calculation of the "true" fibrinogen value of plasma. By this method, "normal" plasma fibrinogen values, as determined with thrombin methods, have an error of  $\approx \pm 20$  per cent. owing to occluded proteins.

J. N. ASHLEY

**2172. The determination of total base of serum with an ion-exchange resin conditioned as iodate.** J. C. Vanatta and I. Cushing (*J. Biol. Chem.*, 1954, **208** [1], 195-204).—A method is described for determination of the total base of serum by use of ashed or un-ashed samples. The sample is passed through a column of IRA-400 resin (hydroxide form) pre-treated with 0.0015  $M$   $KIO_3$ . An aliquot of the eluate is treated with  $NaI$  and aq.  $H_3PO_4$ , and the liberated I is titrated with 0.003  $M$   $Na_2S_2O_3$  using starch as indicator. The quantities used in the method are chosen so that 1 ml of  $Na_2S_2O_3$  represents 10 milli-equivalents of total base per litre of original sample. This iodimetric method can be converted into a colorimetric method; part of the liberated I is removed by means of  $Na_2S_2O_3$ , and the remainder is determined spectrophotometrically at 420  $m\mu$ . Ashing of the serum is effected with  $H_2SO_4$ . With un-ashed sera, the results are 4.8 milli-equivalents per litre lower than those obtained with ashed material.

J. N. ASHLEY

**2173. A simple, new method for the estimation of urea in the clinical laboratory.** F. Bode and U. M. Ludwig (*Schweiz. med. Wochschr.*, 1954, **84** [22], 629-630).—The simple and specific methods for the determination of urea in blood and urine

described are based on a colour reaction with *p*-dimethylaminobenzaldehyde (Barrenscheen and Weltmann, *Biochem. Z.*, 1922, **131**, 591). The procedure for urine analysis is as follows. A 0.02-ml portion of a 1 in 10 dilution of urine is placed on a paper strip. After drying, the paper is passed through a soln. of *p*-dimethylaminobenzaldehyde in HCl and then left in the dark for 16 hr. for the colour to develop fully. A square of paper enclosing the spot is cut out, together with a further square for a blank control. Each square is cut into strips and covered with 6 ml of pure pyridine; the coloured compound is eluted by heating for 1 hr. at 90° C in a water-bath. After cooling, the extinction of the test eluate is measured against that of the blank at 450 m $\mu$ . The corresponding urea value is determined by reference to a calibration chart over a range of 5 to 70  $\mu$ g of urea. The procedure for estimation in serum and blood is the same after the test fluid has been prepared by treating 1 ml of serum with 2 ml of ethanol, centrifuging and applying 0.3 ml of the supernatant liquid to the paper or by treating 100 cu. mm of blood with a small quantity of ethanol, filtering quantitatively and transferring the whole to the paper.

G. W. CAMBRIDGE

**2174. Spectrophotometric method for estimation of coproporphyrins.** R. Kehl and B. Günter (*Klin. Wochschr.*, 1954, **32** [5-6], 121-122).—A quant. method for the determination of coproporphyrin based on absorption in the u.v. range is described. Extinction measurements at 401 m $\mu$  were found to be satisfactory, a linear calibration curve being obtained over the range 0.2 to 6  $\mu$ g. The pH of the solution affects the absorption maximum. Details are given for the preparation of the solution for analysis.

G. W. CAMBRIDGE

**2175. A micro-colorimetric determination of creatine in urine by the Jaffé reaction.** H. H. Taussky (*J. Biol. Chem.*, 1954, **208** [2], 853-861).—The method is based on the complete conversion of creatine into creatinine at  $\approx 100^\circ$  C by dil. picric acid. It is applicable to 10 to 80  $\mu$ g of creatine and the results agree with those obtained by Benedict's method. The urine is treated first with 0.05 N I soln. until a definite colour persists (this eliminates interference due to acetone, acetoacetate and ascorbic acid). After extraction with CHCl<sub>3</sub>, the urine is diluted, and equal amounts are placed in colorimeter tubes for determination of preformed creatinine and in heavy walled centrifuge tubes for determination of total creatinine. The creatine is converted into creatinine by heating with dil. picric acid at pH 2 to 2.5. The final Jaffé reaction is carried out simultaneously in both tubes. The orange-red colour attains greatest intensity in 20 min. and is stable for  $\approx 1$  hr. The intensity of the colour is determined on a photo-electric colorimeter with No. 54 filter, and the amounts of creatine are ascertained from a standard graph. Thymol and toluene have no effect on the determination, and glucose up to 60 g per litre does not interfere under the mild conditions used. A list of substances of biological importance and interest, together with the amounts that do not interfere with the reaction is given.

J. N. ASHLEY

**2176. The determination of creatine and creatinine in urine. A correction factor for the determination of twenty-four hour urinary excretion values.** R. M. Anker (*J. Lab. Clin. Med.*, 1954, **43** [5], 798-801).—

Details are given for carrying out the determinations by means of the Jaffé (picric acid) colour reaction. The creatinine correction factor proposed should be applied when other urinary constituents, such as pregnanediol, are determined on unknown or incomplete (24-hr.) urine specimens.

W. H. C. SHAW

**2177. Simplified method for estimation of 11-oxygenated neutral 17-ketosteroids in urine of individuals with adrenocortical hyperplasia.** A. M. Bongiovanni and G. W. Clayton, jun. (*Proc. Soc. Exp. Biol. Med.*, 1954, **85** [3], 428-429).—11-Oxy-17-ketosteroids (I) and 11-deoxy-17-ketosteroids (II) produce an equal response in the Zimmermann reaction (Z) but the response of I with the Pincus reagent (P) (*Endocrinology*, 1943, **32**, 176) is only about one-half of that of II. In adrenocortical hyperplasia the proportion of I is increased, and a measure of the increase may be obtained by determining the urinary steroids in the same extract with both P and Z and expressing the results as the ratio P/Z. In normal adults P/Z averaged 0.95, range 0.74 to 1.2; untreated individuals with adrenocortical hyperplasia had ratios averaging 0.54, range 0.35 to 0.72, showing a highly significant increase ( $p < 0.01$ ) in excretion of I.

H. F. W. KIRKPATRICK

**2178. The excretion of androsterone glucuronides following testosterone injection.** J. Zander and J. Schmidt-Thomé (*Klin. Wochschr.*, 1954, **32** [1-2], 24-27).—The excretion of pregnanediol in the urine of women after administration of high doses of testosterone propionate has been followed by the methods of Venning (*J. Biol. Chem.*, 1937, **119**, 473; 1938, **126**, 595) and Westphal (*Hoppe-Seyl. Z.*, 1944, **281**, 14). Further identification of androsterones by extraction, chromatographic separation on alumina and identification by infra-red absorption spectrography is described.

G. W. CAMBRIDGE

**2179. Elimination of the fluorescence of oestrogens in urinary extracts by hydrogen peroxide.** C. Heusghem (*Nature*, 1954, **173**, 1043-1044).—Addition of H<sub>2</sub>O<sub>2</sub> destroys the fluorescence given by oestrogens treated by the method of Bates and Cohen (*Endocrinology*, 1950, **47**, 166 and 182) at a rate which varies as concn. of oxidiser. In applying this to urine extracts results are best when 5 per cent. by vol. of 20 per cent. H<sub>2</sub>O<sub>2</sub> is added to the fluorescent soln. Fluorescence due to oestrogens is completely destroyed in 60 min. and subtraction of the residual from the initial fluorescence gives more specific values for urinary oestrogens. The method is neither suitable for non-purified extracts nor applicable to the method of Engel (*J. Biol. Chem.*, 1950, **185**, 255), but when applied to the method previously described by the author (*Annales d'Endocrin.*, 1952, **13**, 479) even better agreement was shown with biological tests than previously reported.

H. F. W. KIRKPATRICK

**2180. Clinical results of liver-function test by loading with Prontosil.** W. Siede and H. Schneider (*Klin. Wochschr.*, 1954, **32** [1-2], 18-20).—A simple liver function test is described that has advantages over other tests; it involves only one intramuscular injection and collection of a 24-hr. urine sample, and the analysis of the sample for the test substance is rapid and specific. Prontosil is excreted as a red substance and its estimation in urine is based on the difference in extinction between a 1 in 10 dilution of

the sample and a similar diluted solution treated with dithionite. The extinction is determined in a step-photometer having a S 50 filter. The concn. of Prontosil is determined from a calibration curve over the range 0 to 16 mg per cent.; hence the total Prontosil excretion can be computed. Comparison with results obtained by the Bromsulphalein (sulphobromophthalein) test shows a higher percentage of positives in liver disease and no false positives in healthy subjects. Presence of bile pigments in urine does not affect the estimation and it is not necessary to control urine volume in any way.

G. W. CAMBRIDGE

**2181. The significance of histidinuria in the differential diagnosis of severe jaundice.** L. Norpoth and E. Ohligschläger (*Dtsch. med. Wochschr.*, 1954, **79** [11], 438-439).—The paper-chromatographic identification of histidine in urine of patients with jaundice is of significance in differential diagnosis, as in liver cirrhosis and parenchymatous jaundice the occurrence of histidinuria is frequent, but positive findings were not attained in cases of obstructive jaundice. The method of separation of histidine is based on that previously described by Norpoth, Clösges and M. Schulze (*Verh. Dtsch. Ges. Verdauungskrrh.*, XVI, Tgg. Essen, 1952; Lubeck, 1953, p. 222). The chromatogram is run in sat. phenol-H<sub>2</sub>O for 12 hr. and then in collidine for 20 hr., the spot being developed with ninhydrin and identified by comparison with a simultaneously run histidine spot.

G. W. CAMBRIDGE

**2182. The determination of zinc with dithizone in biological preparations.** H. Wolff (*Biochem. Z.*, 1954, **325** [4], 267-279).—The dithizone method for the estimation of Zn in biological materials has led to various results. The author has investigated the preparation of material for analysis, the influence of pH on the reaction, the method of estimation of the Zn dithizonate and the preparation of reagents. Care should be taken in the preparation of reagents: (i) distilled water should be freed from metals by ion exchange by passing through Lewatit KSB154 (Bayer), (ii) dithizone should be purified by extracting from a CCl<sub>4</sub> soln. with twice its volume of aq. NH<sub>3</sub> (pH 9 to 11) and removed from the aqueous phase by treatment with dil. HCl (pH 3 to 5) and CCl<sub>4</sub>. With the precautions recommended 76 parallel estimations of normal sera gave a mean of 145.2 µg per cent. with a max. deviation of ± 2.2 per cent. and a mean error for a single estimation of ± 0.7 per cent. Thirty-four estimations on the same erythrocyte suspension gave a mean of 1.01 µg of Zn per 10<sup>9</sup> cells with a max. deviation of ± 3.2 per cent. and a mean error of ± 1.7 per cent. Twenty-two parallel estimations on liver gave a mean of 3.81 µg per g with a max. deviation of ± 3.9 per cent. and a mean error of ± 1.4 per cent.

G. W. CAMBRIDGE

**2183. The photometric micro-determination of cobalt in blood serum and other biological substances.** F. A. Pohl and H. Demmel (*Anal. Chim. Acta*, 1954, **10** [6], 554-561).—Nitroso-R-salt is used as a colorimetric reagent for the determination of 0.02 to 0.3 µg of Co in 5 to 10 ml of blood serum with an accuracy of ± 5 per cent. A special photometer is used with a capillary cuvette 10 cm long and of 0.7 ml capacity. A green filter is used to measure the extinction at 520 mµ.

**Procedure**—Heat the blood sample (10 ml) with a (1 + 1) mixture of conc. HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> (5 ml) and HClO<sub>4</sub> (10 ml) until colourless, and evaporate to dryness. Dissolve the residue in 0.1 N HCl

(50 ml) and adjust to pH 2.5 with aq. NH<sub>3</sub>. Extract Cu<sup>++</sup> by shaking with a 0.02 per cent. soln. of dithizone in CHCl<sub>3</sub> (10 ml) and extract again with CHCl<sub>3</sub> (10 ml). Add a drop of 30 per cent. aq. H<sub>2</sub>O<sub>2</sub>, extract Fe<sup>+++</sup> by shaking with a 1 per cent. soln. of 8-hydroxyquinoline in CHCl<sub>3</sub> (10 ml) and extract 3 times with CHCl<sub>3</sub> (10 ml). Add to the aq. soln. 10 per cent. aq. ammonium tartrate (3 ml) and adjust to pH 5 with dil. aq. NH<sub>3</sub>; add 2 per cent. aq. Na diethyldithiocarbamate (5 ml) and extract 3 times with CHCl<sub>3</sub> (10 ml). Evaporate the extracts to dryness with a surface heater. To the residue add 10 per cent. aq. NaNO<sub>3</sub> (0.2 ml), heat with a (1 + 1) mixture of HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> (0.25 ml) and HClO<sub>4</sub> (1 ml) under a reflux condenser until colourless, and evaporate to dryness. To the residue, add an acetate buffer (pH 6, 0.5 ml) and 0.05 per cent. aq. nitroso-R-salt (0.2 ml) and heat in boiling water for 1 min. Add conc. HNO<sub>3</sub> (0.2 ml), heat again for 1 min. and cool in cold water. Dil. to 1 ml and centrifuge. Transfer 0.7 ml of clear soln. to the capillary cuvette, measure the extinction and interpret the reading from a calibration curve prepared with known amounts of Co.

W. C. JOHNSON

**2184. Determination of methanol in biological fluids by microdiffusion analysis.** M. Feldstein and N. C. Klendshoj (*Anal. Chem.*, 1954, **26** [5], 932-933).—The sample of blood or urine (0.5 ml) is placed in the outer compartment and 2.2 ml of 10 per cent. v/v H<sub>2</sub>SO<sub>4</sub> in the centre cell of a Conway microdiffusion unit; with the unit almost fully covered, 1.0 ml of saturated aq. K<sub>2</sub>CO<sub>3</sub> is injected into the outer cell, which is then covered. After mixing the test fluid, which contains 0.009 to 0.18 mg of methanol, with the K<sub>2</sub>CO<sub>3</sub>, the unit is allowed to stand at room temp. for 2 hr. Into three separate tubes are placed: (a) 1.0 ml of the 10 per cent. v/v H<sub>2</sub>SO<sub>4</sub> from the centre compartment, followed by 1 drop of 5 per cent. w/v KMnO<sub>4</sub>, and, after 5 min. saturated KH<sub>2</sub>SO<sub>4</sub> soln. until the colour is discharged; (b) 1.0 ml of the 10 per cent. H<sub>2</sub>SO<sub>4</sub>; and (c) 1.0 ml of distilled water. Into each of the three tubes are pipetted 0.2 ml of 0.5 per cent. aq. chromotropic acid and 4 ml of conc. H<sub>2</sub>SO<sub>4</sub>. After 15 min. at 100°C, each is cooled and made up to 10 ml. Absorbancy is measured at 580 mµ with reference to the blank (c) and compared with standards prepared similarly. Small amounts of formaldehyde give colours which appear in the liquid from tube (b), and a correction is applied; excessive formaldehyde renders the method inapplicable, but ethanol does not interfere. Results are converted by multiplying by an empirical factor 1.21 to give the true methanol concn. Errors are ± 3 per cent.

D. A. PANTONY

**2185. A simple method for the quantitative estimation of pepsins in gastric juice.** F. Kazmeier and C. Stahlberg (*Klin. Wochschr.*, 1954, **32** [3-4], 85-87).—Compounds of the invert soap type such as Bradosol (domiphen) have marked protein precipitant properties; these have been applied to the estimation of small amounts of protein (Abelin and Pfister, *Brit. Abstr. C*, 1951, 52). The amount of protein remaining after a given period of digestion of a standard egg albumin solution has been used to assess the activity of pepsin in samples of gastric juice. Two soln. are used: (a) 0.25 per cent. egg albumin in glycine buffer (pH 2.0) and (b) 0.2 per cent. of Bradosol in glycine buffer (pH 10.0). A 0.5-ml portion of filtered gastric juice is incubated with 5 ml of (a) for 45 min. at

37° C and 0.5 ml of this mixture is then transferred to a tube containing 5 ml of ice-cold solution (b). The turbidity is estimated in a photometer with a yellow filter (578 mμ). The amount of protein remaining and hence the activity of the pepsin is found by reference to a previously constructed calibration chart for 0 to 0.25 per cent. of egg albumin. Pepsin activity is expressed as percentage decrease of protein. The accuracy of the estimation is given as  $\pm 1.5$  to 2 per cent. The pepsin contents of stomach juice samples in various diseases and under various conditions are reported.

G. W. CAMBRIDGE

**2186. Analysis of hexose phosphates and sugar mixtures with the anthrone reagent.** L. C. Mokrasch (*J. Biol. Chem.*, 1954, **208** [1], 55-59).—The reagent is prepared by dissolving anthrone (1 g) in a cold mixture of  $H_2SO_4$  (d 1.84; 1 litre) and ice (290 g). The solution is stable at 4° C in the dark and remains stable for 2 months. *Procedure for determination of sugars and sugar phosphates*—Keep the reagent (6 ml) in a colorimeter tube in ice-water for 5 min. Add, without mixing, the sample (1 ml) containing < 0.25 micromole of oxohexose, 0.4 micromole of aldohexose, or 1.0 micromole of pentose. Allow to cool, then mix by rapid swirling, and return the tube to ice-water. Close the tube with a capillary vent stopper and heat at  $80^\circ \pm 0.5^\circ C$  for a chosen time. Cool the tube in ice-water (in which it can be kept for several hr.), and determine the colour development spectrophotometrically at 620 mμ. Development curves are given for several carbohydrates and their derivatives. Colour peaks occur after characteristic times of heating with the reagent. Details are given for analysis of mixtures of glucose and fructose, and of glucose and ribose. The method depends on the fact that in a mixture of sugars each sugar requires a definite time of controlled heating for max. colour development.

J. N. ASHLEY

**2187. Determination of protein-bound carbohydrates by the anthrone reaction. Effect of tryptophan.** E. F. Tuller and N. R. Keiding (*Anal. Chem.*, 1954, **26** [5], 875-878).—The cause and effect of the appearance of a 520-mμ absorption peak in the quant. determination of carbohydrate in materials containing protein by the anthrone method was studied. The cause was shown to be due to the formation of a complex between the tryptophan in the protein soln. and the sugar in the presence of excess of anthrone and  $H_2SO_4$ . This complex causes a 5 to 15 per cent. error in the determination, which can be eliminated by preparation of a nomogram formed by plotting the ratio of the absorbances of the 530-mμ peak to the 630-mμ peak of known mixtures against the absorbance of the 630-mμ peak.

G. P. COOK

**2188. Estimation of hexose diphosphate.** J. Kahan (*Arch. Biochem. Biophys.*, 1954, **48** [2], 331-337).—The anthrone reaction is adapted for use with extracts of biological material. *Procedure*—To 1.5 ml of aq. soln. containing 5 to 400 μg of hexose diphosphate in a Pyrex test tube (26 × 200 mm), add 0.5 ml of anthrone reagent (2 per cent. in ethyl acetate) and then a layer of 6 ml of conc.  $H_2SO_4$  under the sample. Swirl gently, then vigorously, until the mixture becomes homogeneous and the anthrone dissolves. Measure the resulting green colour (stable for several hours) at 625 mμ after at least 20 min. against a blank of water and reagents.

Rectilinear calibrations are obtained with the purified Ba salt of hexose diphosphate, with fructose or with glucose as standard; if glucose is used the results are to be multiplied by 1.01.

Corrections are necessary for other carbohydrate-containing compounds (adenosine triphosphate, adenosine diphosphate) extracted under the conditions described.

W. H. C. SHAW

**2189. A new colour reaction for keto [oxo] acids and other carbonyl compounds.** Z. Dische, R. Weil and E. Landsberg (*J. Biol. Chem.*, 1954, **208** [1], 23-28).—α-Oxo-acids, and α-oxo- and hydroxy-aldehydes give a pink coloration when treated with 2-methylindole and HCl. Polyhydroxy-α-oxo acids, polybasic acids, straight-chain fatty acids, amino-acids, sugars, hexuronic acids and glucosone give no colour under the conditions of the reaction. *Procedure*—Add water (0.5 ml) and a mixture (0.5 ml) of equal vol. of 0.2 per cent.  $FeCl_3 \cdot 6H_2O$  and 3 per cent. cysteine hydrochloride and 3 N HCl (3 ml) to a solution (0.5 ml) containing pyruvic acid (1 to 10 μg per ml). Immerse the tube in ice-water and add 0.5 ml of freshly prepared 0.1 per cent. aq. 2-methylindole; shake, and replace the tube in ice-water and keep in a refrigerator for 24 hr. The temp. must not rise locally above 0° C. A pink colour is formed which becomes a max. in 24 hr. The mixture is left at room temp. for a few min., and the optical densities at 494 and 440 mμ are determined spectrophotometrically. The difference between the two values varies as concn. of oxo-acid. For determination in tissues, an extract is prepared with trichloroacetic acid or  $HClO_4$ , and an internal standard is used. A modification which allows detection of glycolaldehyde, triose and oxo-aldehydes in presence of α-oxo-acids is described.

J. N. ASHLEY

**2190. Metabolism of guanidine derivatives. III. Chromatographic analysis.** J. Roche, Ng. van Thoai and J. L. Hatt (*Biochim. Biophys. Acta*, 1954, **14** [1], 71-75).—The separation of guanidine and 11 derivatives (arginine, agmatine, arcaine, arginic acid, taurocyanine, guanidopropionic acid, guanidobutyric acid, methylguanidine, creatine and *asym.*-dimethylguanidine) on paper chromatograms by 10 different solvent systems is described. The position of the substances on the paper is shown with one of three reagent mixtures: (i) a mixture of 0.2 ml each of 40 per cent. NaOH, 40 per cent. urea and 1 per cent. alcoholic 1-naphthol in 10 ml of  $H_2O$ , followed after drying by fresh NaOBr; (ii) a mixture of 10 per cent. sodium nitroprusside, 10 per cent.  $Na_3Fe(CN)_6$  and 10 per cent. NaOH in equal proportions; (iii) a fresh mixture of 0.1 ml of diacetyl and 15 ml of 1 per cent. 1-naphthol in 6 per cent. NaOH. The methods used are applied to show the presence of guanidine, methylguanidine, dimethylguanidine, creatine and arginine in extracts of coelenterates and sponges.

C. E. SEARLE

**2191. Determination of the iodine value of phospholipids.** C. H. Lea and D. N. Rhodes (*Analyst*, 1954, **79**, 304-305).—Results are given of determinations of iodine values of pure fatty esters and phospholipids by the method of Yasuda (*Brit. Abstr. A*, 1932, 185), the Rosenmund-Kuhnemann method (of which the Yasuda method is a micro version) and the Wijs method. These results confirm the findings of other workers that the Yasuda method frequently gives results lower than those of the other methods, and suggest that

there is no reason why the Yasuda method should be preferred to the Wijs (or Hanus) procedure (scaled down to semi-micro quantities when necessary), except, perhaps, for examination of crude lipid extracts of tissues containing sterols in quantity.

A. O. JONES

**2192. Thyroid function and lipid nephrosis.** S. Cruchaud, C. Mahaim, B. Scazziga and A. Vannotti (*Schweiz. med. Wochschr.*, 1954, **84** [17], 478-481).—Thyroid function in two cases of lipid nephrosis has been determined by the usual  $^{131}\text{I}$  technique, and the  $^{131}\text{I}$  excreted in the urine has been detected by a method based upon the butanol extraction of proteins and their subsequent separation by paper chromatography and identification of the  $^{131}\text{I}$ -fractions. The proteins of 200 ml of urine, sampled after the oral administration of  $^{131}\text{I}$ , were pptd. by 10 g of trichloroacetic acid and centrifuged. The ppt. was extracted twice with 15 ml of butanol, which was then evaporated to dryness in the presence of  $\text{H}_2\text{SO}_4$ . The residue was taken up in butanol (1 ml) and, after sedimentation, 0.05 to 0.1 ml of extract was placed on a strip ( $2.5 \times 45$  cm) of Whatman No. 1 paper together with 0.02 ml of thyroxine, di-iodo- and tri-iodothyronine solutions. An ascending chromatogram was run for 30 hr. with pentanol saturated with  $\text{NH}_3$ . The spots were developed with ninhydrin and the radioactivity of the butanol extract fractions was determined with a Geiger-Müller counter.

G. W. CAMBRIDGE

**2193. Investigations on the improvement of the lysozyme estimation method.** N. Burghartz and E. Boosfeld (*Klin. Wochschr.*, 1954, **32** [7-8], 181-182).—The accuracy of lysozyme estimation by the Lobstein and Fogelson method (*Brit. Abstr. C*, 1951, 377) is given as  $\pm 2.5 \mu\text{g}$ . The following modifications are used to attain an accuracy of  $\pm 1 \mu\text{g}$ . The concentration of lysozyme in the test solution (urine) is raised by evaporation at a pressure of 12 mm of Hg at  $37^\circ\text{C}$ . This raises the salt concentration, so the conc. solution must then be dialysed to remove excess of salts. The estimation is carried out at  $40^\circ\text{C}$  and the time of observation of lysis is prolonged to 10 min.

G. W. CAMBRIDGE

**2194. isoButanol-acetic acid-water mixture as a solvent for amino-acids.** K. Dakshinamurti (*Curr. Sci.*, 1954, **23** [3], 89).—isoButanol-acetic acid-water (4:1:5) is compared with n-butanol-acetic acid-water for the separation of amino-acids. The  $R_F$  values of the amino-acids are higher with the former solvent, whilst lysine and histidine, which are non-separable with n-butanol, have distinct  $R_F$  values and are well separated. Distinct and well-defined bands are attained and a useful separation of amino-acids is achieved.

D. BAILEY

**2195. The quantitative determination of proline and pipecolic acid with ninhydrin.** R. S. Schweet (*J. Biol. Chem.*, 1954, **208** [2], 603-613).—The method for determination of proline and pipecolic acid (piperidine-2-carboxylic acid) is based on formation of a red colour when the acids react with ninhydrin in acetic acid under nearly anhydrous conditions. Most primary amino-acids, hydroxyproline and  $\text{NH}_2$  give little or no colour under the given conditions. *Procedure for proline*—Put a solution containing 1.5 to  $10 \mu\text{g}$  of proline into 18  $\times$  150-mm test tubes, and adjust the water vol. to exactly 0.05 ml (either by evaporating to

dryness at room temp. *in vacuo* and then dissolving the residue in 0.05 ml of water or by mixing with acetic acid, so that an aliquot contains the correct vol. of water). Add 0.2 ml of a mixture of conc.  $\text{HCl}$  (1 ml) and acetic acid (99 ml), and then add acetic acid to give a total vol. of 3.85 ml. Finally, add the ninhydrin reagent (0.3 ml) [ninhydrin (372 mg) in acetic acid (20 ml); the solution should be freshly prepared each day], cover the tubes with aluminium caps and heat in a bath of glycerol at  $121^\circ$  to  $122^\circ\text{C}$  with the level of the bath 0.5 in. above the liquid in the tubes. After exactly 5 min., place the tubes in ice, and cool them to room temp. Add sufficient acetic acid to make a final vol. of 4.15 ml, and determine (within 15 min.) the optical density spectrophotometrically at 530  $m\mu$ ; the amount of proline is then ascertained from a standard graph. If pipecolic acid is also present, the optical density is determined at 530 and 560  $m\mu$ , and a formula is given for calculation of the amount of proline. A modification of the method is described for protein hydrolysates. *Procedure for pipecolic acid*—Use an aliquot containing 1.5 to  $10 \mu\text{g}$  with the water vol. adjusted exactly to 0.05 ml. Add acetic acid to make a total vol. of 3.95 ml. Add the ninhydrin reagent (0.2 ml) and proceed as described above except that the tubes are heated for 8 min. The optical density is determined at 560  $m\mu$ ; and a formula is given for use when proline is also present.

J. N. ASHLEY

**2196. The electrophoresis of protein-poor fluids after ultra-filtration through a collodion thimble.** H. Kutzim (*Dtsch. med. Wochschr.*, 1954, **79** [5], 168-170).—For electrophoretic analysis of proteins it is necessary to use a 2 to 6 per cent. w/v solution. Body fluids such as blister and cerebrospinal fluids and urine have been concentrated by ultra-filtration before electrophoresis by the Grassmann technique (Grassmann and Hannig, *Brit. Abstr. C*, 1952, 554). The ultra-filtration was through a collodion thimble under a negative pressure of 200 cm of water for 3 to 4 hr. (original volume 15 ml). Details are given of the preparation of the collodion thimble, the membrane being impermeable to haemoglobin. As a test of recovery, a 1 in 300 dilution of serum was concentrated by this method and the electrophoretic analysis was compared with that of the undiluted serum. The results were in reasonable agreement. Cerebrospinal fluid with a total protein content of 24 mg per 100 ml has been successfully analysed by this method.

G. W. CAMBRIDGE

**2197. The paper-chromatographic separation and qualitative identification of mixtures of  $\text{C}_{19}\text{O}_3$  steroids.** G. Arroyave and L. R. Axelrod (*J. Biol. Chem.*, 1954, **208** [2], 579-589).—The paper-chromatographic separation of twelve  $\text{C}_{19}\text{O}_3$  steroids is described. Whatman No. 1 filter-paper is used; the solvent systems are methylene chloride-formamide, cyclohexene-formamide, and methylcyclohexane-propylene glycol, and the chromatographic procedure is essentially that of Burton *et al.* (*J. Biol. Chem.*, 1951, **188**, 763) with slight modifications. When the positions of the various steroids are detected on a strip by means of alkaline m-dinitrobenzene, 2:4-dinitrophenylhydrazine and conc. fuming  $\text{H}_2\text{SO}_4$ , the corresponding area in the remainder of the chromatogram is eluted with methanol; aliquots of the eluate are used for spectrophotometric determination in methanol and in conc.  $\text{H}_2\text{SO}_4$ . Separation of the following

steroids is described: androst-5-ene-3 $\beta$ :16 $\alpha$ :17 $\beta$ -triol, aetiocolane-3 $\alpha$ :11 $\beta$ :17 $\beta$ -triol, androst-4-ene-6 $\alpha$ -ol-3:17-dione (I), androst-4-ene-11 $\beta$ :17 $\beta$ -diol-3-one (II), androst-4-ene-6 $\beta$ -ol-3:17-dione (III), androst-4-ene-11 $\beta$ -ol-3:17-dione (IV), aetiocolane-3 $\alpha$ :11 $\beta$ -diol-17-one (V), androstane-3 $\alpha$ :11 $\beta$ -diol-17-one, aetiocolan-3 $\alpha$ -ol-11:17-dione, androstane-3:16:17-trione, androst-4-ene-3:11:17-trione (VI) and androstane-3:11:17-trione. The two triols are widely separated in the system methylene chloride-formamide, and thus epimers of these may be separated with this system as they become available. Compounds IV and V are not separated by the cyclohexene-formamide system, but are readily separated by means of methylcyclohexane-propylene glycol. Compounds that contain the  $\Delta^4$ -3-oxo group give an orange colour with 2:4-dinitrophenylhydrazine, whilst 3-oxo compounds without conjugated unsaturation give only a yellow colour. With alkaline *m*-dinitrobenzene, the 17-oxosteroids give a purple colour, while 3-oxosteroids give a blue colour. The triols give no colour with either reagent; the first-named triol gives a green colour with conc.  $H_2SO_4$ , whilst a deep red colour is obtained with the second triol. I, II, IV, III and VI have  $\lambda_{max}$  at 239.5, 242, 240, 235.5 and 238  $m\mu$ , respectively. Absorption spectra of the  $H_2SO_4$  chromogens are given. J. N. ASHLEY

2198. The fluorescence reactions of steroids. J. W. Goldzieher, J. M. Bodenchuk and P. Nolan (*Anal. Chem.*, 1954, **26** [5], 853-856).—Twenty-nine steroids were investigated, each being treated with  $H_2SO_4$ ,  $H_3PO_4$  and formic acid and the resulting fluorescence being measured. Results indicated that the product resulting from heat treatment of the steroid in conc.  $H_2SO_4$  gives max. fluorescence in most instances when irradiated with 436- $m\mu$  light. Generally, the greater the number of hydroxyl groups the greater is the fluorescence energy,  $\alpha$ -hydroxyl groups being more fluorogenic than  $\beta$ -groups. Steric molecular configuration affects both the characteristics and intensity of the spectrum. Presence of an aromatic A-ring also greatly increases fluorescence. The results obtained should be of considerable value in the elucidation of steroid molecular structures by fluorimetry and probably provide the basis for the development of analytical methods. G. P. COOK

2199. The role of the liver in the excretion and breakdown of adrenal cortical hormones. K. W. Brückel, H. J. Hübener, G. Meyerheim and G. Liersch (*Klin. Wochschr.*, 1954, **32** [1-2], 21-24).—The method for the chromatographic analysis of adrenal cortical steroids in urine described is carried out as follows. A 350-ml sample from a 24-hr. urine specimen is brought to pH 1.1  $\pm$  0.1 with HCl and is then subjected to continuous ether extraction for 48 hr. The ether is removed *in vacuo* at 45°C, and the residue is taken up in 150 ml of  $CHCl_3$ , washed 4 times with 0.1 N NaOH and three times with 25 ml of 0.1 N HCl and  $H_2O$ . The  $CHCl_3$  is removed *in vacuo* at 45°C and the residue (in methanol) is transferred to a 19  $\times$  38-cm sheet of paper. An ascending chromatogram is run overnight with a (1 + 3) mixture of methanol and  $H_2O$ . The position of the spots is determined by contact photography in u.v. light, the material with an  $R_F$  value of 0.78 being the  $M_2$  fraction (Hübener, Meyerheim and Brückel, "Sympos. über Probleme des Hypophysen-Nebennieren-Systems," 1952, p. 101). This section is cut out and eluted by descending ethanol for 3 days. The eluate contains the free

adrenal cortical hormones; the alcohol is removed *in vacuo* at 45°C and the residue is taken up in chloroform and transferred to 2.4-cm  $\times$  4-cm strips. Descending chromatograms are run for 80 hr. with toluene-propylene glycol (Zaffaroni, Burton and Keutmann, *Science*, 1950, **111**, 6). The  $\Delta^4$ -3-one steroids are indicated by u.v. light photography and the corresponding spots are eluted and examined spectrometrically. Absorption at 240  $m\mu$  can be used for quant. determination. G. W. CAMBRIDGE

2200. Further simplification and improvement of the determination of corticosteroids by paper chromatography. H. Schmidt and H. Staudinger, with V. Bauer (*Biochem. Z.*, 1953, **324** [2], 128-133).—The method formerly described (Hofmann and Staudinger, *Biochem. Z.*, 1951, **322**, 230) can be improved especially for adrenal extracts by using water-heptanol mixtures as solvent and a special paper (Schleicher and Schüll, 2043 b) for the chromatogram. The time taken for the chromatography has been reduced from 30 to 9 hr., and the quant. determination by spraying with triphenyltetrazolium chloride has been simplified and improved. A. J. MEE

2201. The use of pyrophosphate buffer for the manometric assay of xanthine oxidase. S. B. Dhungat and A. Sreenivasan (*J. Biol. Chem.*, 1954, **208** [2], 845-851).—Pyrophosphate inhibits the endogenous respiration of rat-liver homogenate, but it has no effect on xanthine oxidase, and the enzyme can be determined manometrically by use of xanthine as oxidisable substrate in pyrophosphate buffer at pH 8.6. The results agree with those obtained from determination of the disappearance of xanthine when added to the buffer mixture. With pyrophosphate buffer there is 20 to 25 per cent. inhibition of uricase activity of rat-liver homogenate, but oxidation of urate is much more rapid than that of xanthine, and in no way limits oxidation of xanthine. J. N. ASHLEY

2202. On the determination of xanthine oxidase activity in animal tissues. L. S. Dietrich and E. Borries (*J. Biol. Chem.*, 1954, **208** [1], 287-292).—A very sensitive colorimetric method is described for determination of xanthine oxidase in mouse tissues. The method readily distinguishes xanthine oxidase from uricase activities, and is applicable to tissues of low xanthine oxidase activity. It is adaptable to large-scale and routine use and requires no special apparatus. The tissue is homogenised in 0.039 N phosphate buffer (pH 7.3), and an aliquot is incubated with xanthine at 37°C with constant agitation. Samples are withdrawn for determination of uric acid and allantoin at regular intervals; uric acid is determined by the method of Brown (*Brit. Abstr. C*, 1945, 248) and allantoin by a modification of the method of Young *et al.* (*Brit. Abstr. A III*, 1942, 503; *Brit. Abstr. C*, 1944, 174). Owing to the abundance of uricase in liver, liver xanthine oxidase is determined by measuring allantoin formation as a function of time. With kidney, heart, lung and testis, which have no measurable uricase activity, analyses for uric acid are adequate as a measure of xanthine oxidase activity. For small intestine, determinations of both uric acid and allantoin must be carried out. J. N. ASHLEY

2203. Phototurbidimetric method for determination of lipase in canine pancreatic juice. A. Grossberg, P. Guth, S. Komarov and H. Shay (*Rev.*

*Canad. Biol.*, 1953, **12**, 495-508).—A turbidimetric method is described for determining lipase in pancreatic juice. Vegetable oil emulsion (0.02 per cent.) is used as substrate in a phosphate buffer pH 7.0, ionic strength 0.15, and containing 3 per cent. of gelatin and 0.004 per cent. of potassium oleate as activators and stabilisers for the enzyme. The reaction is neither of zero nor of first order over the whole range, but a linear relationship exists over a limited range and is expressed as  $L.U. = 1.22 \times K_0^{0.637}$  where  $K_0$  is the zero-order velocity constant. One lipase unit (L.U.) is defined as the lipase activity contained in that amount of freshly secreted canine pancreatic juice (postprandial, after a meal of lean meat) containing one  $\mu$ g of protein nitrogen. E. C. BUTTERWORTH

2204. Enzyme systems of the silk-worm, *Bombyx mori* Linn.: Part II. A paper-chromatographic micro-method for the detection and characterisation of peptidases. B. Bheemeswar and M. Sreenivasaya (*J. Sci. Ind. Res., B, India*, 1954, **13** [3], 191-194).—A simple paper-chromatographic micro-method for the determination of peptidases is described. The enzyme solution acts on a peptide substrate in phosphate buffer of pH 7.2, and the amino-acids are separated on a filter-paper with butanol-acetic acid-water (10:2.5:10) as solvent. The amino-acids are detected by u.v. or by spraying the paper with ninhydrin. The method has a precision comparable with that of the Linderstrom-Lang ultra-micro method and will reveal side-reactions, such as trans-aminations and trans-peptidations, in the reaction mixture. It has been applied successfully to the study of peptidase activity of the tissues and body fluids of the silk-worm, *Bombyx mori* Linn. G. C. JONES

2205. Analyses of biological materials as indexes of exposure to organic solvents. H. B. Elkins (*Arch. Ind. Hyg.*, 1954, **9** [3], 212-222).—Existing methods of estimating human exposure to solvents by analysis of blood or urine for the unchanged solvents or their metabolites are reviewed. Urine analysis is valuable for determining unchanged methanol and metabolites of methyl acetate, toluene, trichloroethylene and aniline (methanol, hippuric acid, trichloroacetic acid and diazotisable substances, respectively). Urine sulphate ratios are determined in cases of exposure to benzene vapour. Blood bromide determinations are carried out following exposure to organic bromides. It is suggested that analyses of bromide and carbon disulphide and possibly carbon tetrachloride in urine would be useful. C. E. SEARLE

See also Abstracts 2054, 2292.

### Drugs

2206. Determination of alkaloids by the reaction of Caille and Viel with antimony iodide. S. Besson and J.-J. Brignon (*Ann. Pharm. Franç.*, 1953, **11** [7-8], 535-540).—A rapid sensitive method for the quantitative determination of alkaloids is described based on the reaction of Caille and Viel (*Compt. Rend.*, 1923, **176**, 1156). The alkaloid in dil. HCl soln. is pptd. with a 1 in 5 dilution of a soln. containing 5 g of  $SbCl_3$  or  $Sb_2O_3$ , 20 ml of conc. HCl and 40 g of KI in 100 ml of water. The ppt. is removed by centrifuging, and the excess of reagent is determined in the decanted liquid. The ratio of  $SbI_3$  to alkaloid has been determined for aconitine, atropine, brucine, caffeine, emetine, morphine, quinine, sparteine and strychnine. N. M. WALLER

2207. Chromatographic determination of codeine in opium and other complex mixtures. G. C. McElheny, G. DeLaMater and R. D. Rands (*Anal. Chem.*, 1954, **26** [5], 819-823).—Opium is extracted with a saturated soln. of sodium acetate and the non-phenolic alkaloids are isolated by extraction in benzene solution. Separation is carried out on an alumina column by means of developing solutions containing isopropanol,  $CHCl_3$  and benzene. Thebaine, cryptopine, neopine, papaverine and narcotine are collectively eluted before codeine. The codeine content of the eluate fractions is determined by titration with standard acid, after evaporation and solution of the fraction in methanol. The method gives results that deviate from the true value by less than 2 per cent. G. P. COOK

2208. Assay of Tetraon [papaveretum]. C.-G. Lindblad and A. Ågren (*Farm. Revy.*, 1954, **53** [4], 69-79).—The determination by means of partition chromatography of the alkaloids codeine, morphine, papaverine and narcotine in papaveretum is described. The stationary phase is a phosphate buffer. To 0.4 g of papaveretum in 20 ml of  $H_2O$  are added 5 ml of 0.2 N NaOH, and the mixture is extracted twice with a mixture of 10 ml of  $CHCl_3$  and 30 ml of ether, and then with 10 ml of  $CHCl_3$ . The filtered extracts are evaporated to 0.5 to 1 ml, and diluted with 25 ml of ether before being transferred to the prepared column. The alkaloids are eluted in the order: narcotine (with 200 ml of ether saturated with  $H_2O$ ), papaverine (150 ml of  $CHCl_3$ ) and codeine (250 ml of  $CHCl_3$  saturated with  $NH_3$ ), and are then determined colorimetrically. N. M. WALLER

2209. The polarographic determination of cephaeline. A. Jindra, V. Jungr and J. Zýka (*Acta Pharm. Int.*, 1953, **2** [5], 397-405).—Cephaeline can be determined in the presence of a considerable excess of emetine by converting it to the nitroso derivative with  $NaNO_2$  and HCl at 20° C for 10 min., adding methanol to keep the emetine in solution, then an excess of KOH in which the nitroso derivative is stable, removing the O in a stream of N and polarographing. The K salt of the nitroso derivative of cephaeline in an alkaline medium is reduced at a potential of -0.9 V vs. the S.C.E. Results show an accuracy of about  $\pm 3$  per cent. on 5 to 100 mg per cent. of cephaeline. Results for cephaeline in ipecacuanha root were 5 to 10 per cent. higher than those given by a direct-titration method. F. R. MUMFORD

2210. Polarographic determination of cephaeline. O. Gry (*Acta Pharm. Int.*, 1953, **2** [5], 383-395).—Cephaeline, after isolation as in the pharmacopoeial assay, on nitrosation ( $KNO_3$ -HCl), keeping for 5 min., and addition of KOH and tylose gives a solution suitable for polarographic estimation, a straight line graph being given for mean step height plotted against concn. of hydrochloride in the range 1 to 10 mg per 5 ml. The max. deviation is  $\pm 5$  per cent. In the concn. range 0.1 to 1 mg per 5 ml, the presence of twice the amount of emetine does not affect the result. With more concentrated solutions the emetine is precipitated in alkaline solution, but filtration does not give reproducible results. F. R. MUMFORD

2211. Paper-chromatographic separation and fluorimetric determination of cardiac glycosides and aglycones from *Digitalis purpurea*. K. B. Jensen (*Acta Pharmacol. Tox. Kbh.*, 1954, **10** [1],

69-82).—The qualitative chromatographic separation (*Acta Pharmacol. Tox., Kbh.*, 1953, **9**, 99) and the fluorimetric procedure previously described (*Acta Pharmacol. Tox., Kbh.*, 1952, **8**, 101 and 1953, **9**, 66) are modified for the determination of purpurea glycosides and aglycones of both the A and B series. With the apparatus and fluorimeter used, fluorescence curves are linear for digitoxin (2 to 15  $\mu$ g), for gitoxin (1 to 10  $\mu$ g) and for equimolecular amounts of the other compounds. The estimated standard deviation is  $\pm 2$  to 6 per cent. according to the amounts used. W. H. C. SHAW

**2212. Glycosides and aglycones. CXXVIII. Paper chromatography of strongly polar cardiac glycosides and aglycones.** E. Schenker, A. Hunger and T. Reichstein (*Helv. Chem. Acta*, 1954, **37** [3], 680-685).—A suitable method for the paper chromatography of strongly polar glycosides is described. Paper soaked in water serves as stationary phase and *n*-butanol or *n*-butanol-toluene as mobile phase. H. WREN

**2213. Physical methods for the identification of narcotics. IA. Introduction.** C. G. Farmilo and Leo Levi (*Bulletin on Narcotics, United Nations*, 1953, **5** [4], 20-27).—A review is given of the physical methods that can be applied to the identification of narcotics. The methods include crystallography, X-ray diffraction, optical rotation, spectrography, infra-red, ultra-violet and Raman spectra, electro-metric titration and polarography. A table shows some chemical groups occurring in narcotics and the physical methods by which they are most likely to be detected. N. E.

**2214. Physical methods for the identification of narcotics. IB. Common physical constants for identification of ninety-five narcotics and related compounds.** C. G. Farmilo, P. M. Oestreicher and Leo Levi (*Bulletin on Narcotics, United Nations*, 1954, **6** [1], 7-11).—The 95 narcotics and related compounds listed were obtained mainly from commercial sources. A number of free bases were prepared from the salts. Results for water content, melting or boiling-point and specific rotation are given. N. E.

**2215. The use of "Kunstlichen Höhensohne—original Hanau" [u.v. light] in qualitative analysis. III. Detection of barbituric acid, 2-thiobarbituric acid and pyridine.** H. Freytag (*Z. anal. Chem.*, 1954, **142** [1], 12-15).—Pyridine in 20 per cent. alcoholic solution spotted on filter-paper decomposes under u.v. light to give the ammonium salt of enol glutaconic aldehyde; this reacts with barbituric and thiobarbituric acids to give lilac polymethine dyes. The reaction is a sensitive spot test for these compounds and can be used conversely to detect pyridine, but is then less sensitive. P. S. STROSS

**2216. The chromatographic analysis of mixtures of 2-phenylquinoline-4-carboxylic acid [cinchophen] and salicylic acid.** A. Castiglioni and M. Vietti (*Z. anal. Chem.*, 1954, **142** [1], 15-17).—A qualitative study of the chromatographic behaviour of cinchophen and salicylic acid with various adsorbents and eluting soln. is made. Columns of basic alumina give good separation both by ascending and descending techniques with water-saturated butanol as eluting soln., the salicylic acid being strongly adsorbed and the cinchophen passing

quickly through the column. Paper chromatography also gives good separation. With water-saturated butanol, the  $R_F$  values for cinchophen and salicylic acid are 0.7 and 0.5, respectively.

P. S. STROSS

**2217. Determination of camphor in spirit of camphor. Extraction with di-(2-chloroethyl) ether.** E. M. Plein and C. F. Poe (*Acta Pharm. Int.*, 1953, **2** [5], 407-412).—Polarimetric examination of a number of solutions of camphor in di-(2-chloroethyl) ether shows that the instrument reading gives a straight line graph when plotted against percentage w/v of camphor. In the proposed assay, the spirit of camphor is mixed with twice its vol. of 10 per cent. Pb acetate soln. (to ppt. the camphor and reduce its solubility in the aq. ethanol layer) and extracted 3 times with di-(2-chloroethyl) ether; the resulting extract is again shaken with Pb acetate soln., which is extracted twice with fresh solvent. The organic solvent extracts are combined, made up to volume and filtered if necessary, and the polarimeter reading is taken. By reference to a suitable table or graph constructed from standard solutions, the percentage of camphor in the spirit can be calculated. Test analyses give results accurate to within 0.1 per cent. F. R. MUMFORD

**2218. A rapid method for the determination of the ethanol content of tinctures, alcoholic preparations, spirits, wine, beer and must. [Parts I and II.]** R. Fischer and F. Kolmayr (*Pharm. Zentralh.*, 1954, **93** [2], 54-61; [3], 87-92).—The method described is rapid (15 to 20 min.) and has been used satisfactorily for more than 30 tinctures and many other alcoholic preparations and beverages. Results compare favourably with those obtained by the distillation method, the error being 0.1 to 0.2 per cent. for low ethanol concn. and 0.5 to 1 per cent. for high concn. Five ml of tincture are diluted with 10 ml of H<sub>2</sub>O or aq. soln. of precipitant (Pb acetate, NaOH, etc.), mixed and filtered. Five ml of the filtrate are saturated with K<sub>2</sub>CO<sub>3</sub> and after 1 to 2 min. are extracted with 2.5 ml of benzene. The resulting emulsion is centrifuged until sufficient clear liquid is available for the determination of the refractive index of the ethanol-benzene mixture. The ethanol content is read from one of the prepared graphs. Slight modifications of the preliminary pptn. are necessary according to the nature of the sample. N. M. WALLER

**2219. Comparative study of the determination of moisture in certain French Pharmacopoeia products by the Karl Fischer method and oven or vacuum drying.** L. Domange and S. Longueval (*Ann. Pharm. Franç.*, 1953, **11** [7-8], 530-535).—Moisture contents determined by the Karl Fischer method and by oven or vacuum drying are compared for over 100 products. Erroneous results are obtained by the Fischer method for As compounds, Bi and Li salts, CuSO<sub>4</sub> and Cu acetate, ZnO, MgO and MgHCO<sub>3</sub>, and some Na salts. N. M. WALLER

**2220. Determination of isopropanol in dextran and dextran solutions.** G. J. Frisone (*Anal. Chem.*, 1954, **26** [5], 924-925).—The method is based on the oxidation of isopropanol to acetone by K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in H<sub>2</sub>SO<sub>4</sub> soln. Excess of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> is removed by adding NaOH and the acetone is distilled into hypiodite soln. The excess of I then remaining is titrated with standard Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and the percentage of isopropanol is calculated. The average error is < 1 per cent. G. P. COOK

**2221. Quantitative method of determination of oxygen dissolved in antibiotic culture liquids.** B. P. Bruns, E. M. Savitskaya and T. S. Petrova (*J. Anal. Chem., U.S.S.R.*, 1954, 9 [1], 42-46).—Apparatus for taking samples and for polarographic determination of O in solutions containing penicillin or streptomycin are described. G. S. SMITH

**2222. Polarometric [amperometric] titrations in pharmaceutical analysis and drug control.** R. Kalvoda and J. Zýka (*Acta Pharm. Int.*, 1953, 2 [5], 365-382).—A review is given of the methods and applications of amperometric titration in pharmaceutical analysis with 56 references.

F. R. MUMFORD

**2223. Routine pyrogen testing.** K. L. Smith (*J. Pharm. Pharmacol.*, 1954, 6 [5], 309-316).—The author describes the routine testing in rabbits of samples for freedom from pyrogenicity. Both sexes of the Belgian hare breed (2.5 to 4 kg) are used, temperatures being taken per rectum and samples being injected intravenously.

The procedure adopted differs in several respects from that recommended in the B.P. (1953): (i) a constant volume of solution (approx. 30 ml) is injected irrespective of body weight (B.P. specifies 10 ml per kg); (ii) the solution is injected at room temperature instead of at 37° C; (iii) two rabbits (instead of three) are used for each sample—the sample is passed as non-pyrogenic if the temperature rise of each rabbit is < 0.6° C and the mean < 0.5° C; if not, a further two rabbits are used, and if the mean rise of all four is < 0.6° C the sample is passed—(iv) in order to reduce variables to a minimum, solutions are not made isotonic with pyrogen-free NaCl as allowed in the B.P. (1953).

The use of clinical thermometers ( $\frac{1}{2}$  min.) for temperature measurement was found to be quite satisfactory provided the restraint of the rabbit is kept to a minimum during the measurement. Thermocouples are more convenient for routine work; the apparatus is a copy of that in use by the Wellcome Foundation before the incorporation of a recording galvanometer. The animals are restrained in boxes and thermocouples are inserted 4 in. into the rectum and secured by tying them to the tail. The first temperature is taken 30 min. later, then at 20 min. intervals throughout the test. The injection time is noted and the temperature rise is determined by the difference of the highest temperature recorded in the 3 hr. after injection and the mean of the three temperature readings before injection.

The author suggests the use of thermistors as described by Williams and Thompson (*Science*, 1948, 108, 90), for the determination of rectal temperatures. G. B. CHESHER

See also Abstract 2170.

#### Food

**2224. Kjeldahl determination of nitrogen: a critical study of digestion conditions—temperature, catalyst and oxidising agent.** H. A. McKenzie and H. S. Wallace (*Aust. J. Chem.*, 1954, 7, 55-70).—The effect of temp. on the rate of Kjeldahl digestion in the absence of catalyst or oxidising agent has been studied. Both the clearing time and the min. time for complete recovery of N are markedly decreased by raising the digestion temp. with  $K_2SO_4$ . The appreciable rise in temp. during prolonged digestions (due to loss of  $H_2SO_4$ ) and the effect of time and temp. on the pyrolytic loss of N

are considered. By proper choice of digestion conditions, N can be completely recovered in a reasonable time even from refractory compounds, e.g., tryptophan. The time may be further reduced by the use of Hg as catalyst. The use of  $H_2O_2$  as an oxidant is discussed; it is found that earlier claims regarding complete recoveries of N with few additions of  $H_2O_2$  cannot be substantiated. A modified apparatus for the distillation of  $NH_3$  from Kjeldahl digestions is described and acidimetric methods for the determination of the  $NH_3$  are critically examined. The recommended procedure is as follows. The sample containing 0.3 to 1.0 mg of N is placed in a 30-ml micro-Kjeldahl flask, and 1.5 ml of 36 N  $H_2SO_4$ , 1.5 g of  $Na_2SO_4$  and 0.5 ml of a solution (prepared by dissolving 10 g of red HgO in 4 N  $H_2SO_4$  and diluting to 100 ml) are added. The  $H_2O$  is boiled off, and the mixture heated for 15 min. after clearing. The  $NH_3$  is steam-distilled after an NaOH- $Na_2S_2O_3$  solution has been added, absorbed in boric acid, and titrated with  $KH(IO_3)_2$  by using methyl red - methylene blue (2 + 1) as indicator. L. VALENTINE

**2225. Determination of reducing sugar. VI. Determination of invert sugar by means of a modified Blom's method.** Y. Matsuo and H. Mori (*J. Ferment. Technol.*, 1954, 32 76-79).—The invert sugar factor determined by the modified method (see *Anal. Abstr.*, 1954, 1, 763) was 15.40. The invert sugar concn. (mg per 100 ml) is then 1540/titre in ml. The method is accurate to  $\pm 1.6$  per cent. within the range 65 to 165 mg per 100 ml. Total sugars and direct reducing sugars were determined by this method in Japanese black sugar, cane molasses, jam, marmalade and squash; the values found were 0.1 to 2.8 higher than those given by Lane's method. SUGAR IND. ABSTR.

**2226. Studies on raw sugars. I. Analysis of raw sugars. II. Determination of the affination number.** M. Kamoda (*Proc. Res. Soc. Japan Sugar Refineries Technologists*, 1953, 2, 6-9, 10-17).—I. Analytical figures for 29 Cuban and 12 Formosan raw sugars are tabulated. The only significant difference was in colour, the value for Cuban sugar being 38 per cent. higher than that for Formosan sugar. II. A new affination test was devised as follows. Mix 100 g of raw sugar with 45 ml of water, stir for 15 min. at 20° C and centrifuge. Adjust the syrup to pH 7.0 and 67° Brix and use this as washing liquor, pouring 37 g over 100 g of raw sugar sample at 20° C, mixing for 15 min. and then centrifuging in a 6-in. diam. centrifuge for 10 min. at 2000 r.p.m. Determine the pol. and colour value of the washed sugar. The affination number is  $(100 - PY) \times (C - 2)$  where PY and C are pol. and colour (° Stammer), respectively. Results are given for some Cuban, Formosan and Brazilian sugars; the Cuban sugars had a far higher affination number and were much inferior in quality. SUGAR IND. ABSTR.

**2227. Developments in international methods of sugar analysis.** F. W. Zerban (*Sugar, N.Y.*, 1954, 49 [5], 51-53).—This lecture indicates progress to date towards the establishment of internationally agreed methods for sugar analyses. S. C. JOLLY

**2228. Use of Herles' reagent for clarification instead of Horne's dry lead.** H. C. Yu (*Sugar, N.Y.*, 1954, 49 [5], 42-44).—Horne's dry lead (I) was superior to Herles' dry reagent [cryst.  $Pb(NO_3)_2$ , 10 g, and anhydrous  $Na_2CO_3$ , 1.45 g] (II) for clarifying Taiwan molasses after carbonation and defecation processes. For sugar solutions of lower purity,

polarimeter readings of clarified filtrates given by I were higher than readings of those given by II. Clarification and polarisation were affected by the amount of clarifying agent used, especially in crude solutions. Na acetate and  $\text{NaNO}_3$  can both affect polarisation, but the decrease is usually negligible; there was no correlation between salt concn. and depression of rotatory power. S. C. JOLLY

**2229. Creta praeparata as a source of iron in flour.** H. V. Hart (*Analyst*, 1954, **79**, 305).—The Fe content of 8 samples of creta praeparata determined by the method of Pringle (*Brit. Abstr. C*, 1947, 233) for cereals ranged from 57 to 91 mg per 100 g. Added to flour at the statutory rate of 14 oz per 280-lb sack, creta praeparata may thus increase the Fe content of flour by  $\approx 0.25$  mg per 100 g. As the natural Fe content of a 70 per cent. extraction flour is 1.65 mg per 100 g, the creta praeparata addition could supply a third of the amount of Fe required to raise the natural content to the min. statutory level. A. O. JONES

**2230. The determination of creatinine in milk and its application to the investigation of urine therein.** J. Ron Noya and A. Charro Arias (*An. Bromatologia*, 1954, **6** [1], 5-20).—Colorimetric estimation of the creatinine content of 100 samples of cow milk gave values between 0.6 and 2.1 mg per 100 ml. Samples to which creatinine had been added were also studied. The presence of even 1 per cent. of urine is shown to result in a considerable elevation of creatinine content that is readily detectable by the colorimetric method used, viz., a modified van Slyke method ( $\text{CaO}$ ,  $\text{CuSO}_4$  and picric acid) in which ammonium oxalate is used to eliminate excess of Ca, a pH of 7 to 8 is maintained and the picric acid is carefully purified. M. TADMAN

**2231. A method for the detection of foreign fats in dairy products.** V. R. Bhalaria and F. A. Kummerow (*J. Dairy Sci.*, 1954, **37** [2], 156-161).—A method is described for the detection of  $< 10$  per cent. of vegetable or animal fats in butter. To 10 g of butter fat, 100 ml of hot absolute ethanol is added, the clear mixture is cooled to room temp., maintained at  $20^\circ\text{C}$  for 2 hr. and filtered. After washing with 10 ml of absolute ethanol, the residue is dried on a water-bath and then *in vacuo* and its  $n_D^{40}$  is determined. A 10-ml aliquot of the combined filtrate and washings (A) is freed from solvent and the wt. and  $n_D^{40}$  of the dried residue is determined. To a further 75-ml aliquot of A, 5 ml of a 0.001 per cent. Na methoxide solution (4.3 g of Na dissolved in absolute methanol, cooled and diluted to 1 litre with methanol; 1 ml is diluted to 1 litre with ether when required) are added, and after 1 hr. the mixture is neutralised to phenolphthalein with dil.  $\text{H}_3\text{PO}_4$  (0.71 g of 85 per cent.  $\text{H}_3\text{PO}_4$  in 1 litre of water); 10 ml of water are added and the unesterified triglycerides are allowed to settle. A 50-ml aliquot of the clear soln. mixed with 50 ml of ether is extracted with 50 ml of water, and the aqueous phase is discarded. After washing the ethereal phase with water, drying with anhydrous  $\text{Na}_2\text{SO}_4$  and removing the solvent, the  $n_D^{40}$  of the residue is determined. The presence of 10 per cent. of adulterating fat other than coconut oil raises the  $n_D^{40}$  of the alcohol-insol. fraction from  $1.4542 \pm 0.00002$  for genuine butter to  $1.4550 \pm 0.00003$ . Addition of 10 per cent. of coconut oil to butter lowers the  $n$  of the alcohol-sol. fraction from  $1.4540 \pm 0.00002$  to  $1.4532 \pm 0.00005$ . Adulteration also affects the proportion of the three fractions. S. C. JOLLY

**2232. A modified peroxide test for detection of lipid oxidation in dairy products.** C. M. Stine, H. A. Harland, S. T. Coulter and R. Jenness (*J. Dairy Sci.*, 1954, **37** [2], 202-208).—A sensitive and reproducible method for the control determination of peroxide val. is described. To 25 ml of fluid milk, 9 ml of cream or condensed whole milk or 8 g of dry whole milk dispersed in 15 ml of water, in a 9-g 50 per cent. cream test bottle, sufficient BDI reagent (30 g Triton X-100, a non-ionic surface-active agent, and 70 g of Na tetraphosphate in 1 litre of water) is added to bring the vol. to within  $\frac{1}{4}$  in. of the base of the neck, and the bottle is heated for 4 min. in boiling water. More reagent is added to half-fill the graduated portion of the neck of the bottle. After 3 min. longer in the water-bath, the bottle is centrifuged for 1 min., and then heated for a further 3 min. The peroxide val. of the liberated fat is determined by the ferric thiocyanate method of Hills and Thiel (*J. Dairy Res.*, 1946, **14**, 340) with slight modifications. S. C. JOLLY

**2233. Methods for the chemical analysis of ice-cream.** British Standards Institution (B.S. 2472: 1954, 20 pp.).—Standards are laid down for the preparation of the sample, and the determination of total solids, fat, sucrose and reducing sugars, N, ash, Ca and P. The total solids are to be determined by drying on sand; for this a suitable dish, with cover, is described. The Röse-Gottlieb method is to be regarded as the standard method for the determination of fat, where applicable, but for the custard-ice type of sample it is necessary to use the modified Werner-Schmidt method. Sucrose and reducing sugars are determined by titration with Fehling solution, which is standardised against sucrose solution. N is determined by the Kjeldahl procedure by means of Hg, HgO or  $\text{CuSO}_4$  as catalyst. The ash, which is obtained by ignition at  $550^\circ\text{C}$ , is retained for the determination of Ca (oxalate method) and phosphorus (ammonium molybdate and quinol). G. B. THACKRAY

**2234. The analysis of whipped cream containing sugar.** F. T. van Voorst (*Chem. Weekbl.*, 1954, **50** [21], 373-374).—To 40 to 50 g of the cream, add 1 to 2 ml of 5 per cent. Teepol soln. and shake well to homogenise. **Fat determination**—Transfer a weighed sample (2.5 to 3.0 g) to a Gerber-van Gulik cheese butyrometer; add 10 ml of dil.  $\text{H}_2\text{SO}_4$  (sp. gr. 1.5) and heat to  $65^\circ\text{C}$  for 1 hr., add 1 ml of pentanol, shake and make up with dil.  $\text{H}_2\text{SO}_4$ . Centrifuge for 15 min. (1200 r.p.m. in a 40-cm diameter machine), transfer to a water-bath at  $65^\circ\text{C}$  and calculate the fat content. **Sugar determination**—To 5 g of the emulsion washed into a 100-ml flask, add 1 ml of Carrez reagent I and 1 ml of reagent II; make up to vol., mix and filter. Transfer 50 ml of the filtrate to a 100-ml flask, add 5 ml of 30 per cent. HCl soln. and heat to  $68^\circ$  to  $70^\circ\text{C}$  for 10 min. After cooling, add 4 N NaOH soln. to make the reaction neutral to methyl red or methyl orange; make up to vol. and mix. Determine the fructose in 25 ml of this solution by the Luff-Schoorl method, adding 5 drops of liquid paraffin to prevent frothing. In both determinations a correction is made for the Teepol added. E. HAYES

**2235. Determination of fat in lecithin-containing products.** M. E. Stas and A. M. Rohof (*Chem. Weekbl.*, 1954, **50** [21], 372-373).—In determining fat in lecithin-containing foodstuffs, preliminary

boiling with acid to destroy protein causes low results owing to decomposition of the lecithin. An improved method is described for determining the fat in egg-containing products. The sample is mixed with sand, the protein is pptd. by ethanol, and the mixture is evaporated to dryness. After drying the residue at 102° to 105° C, the fat is extracted in a Soxhlet apparatus with  $\text{CHCl}_3$ .

S. C. JOLLY

**2236. The determination of O:O-diethyl O-p-nitrophenol thiophosphate [parathion] residues in tomatoes.** R. Buckley and J. P. Colthurst (*Analyst*, 1954, **79**, 285-289).—A method is described for the separation and determination of parathion residues in tomatoes. The surface of the fruit is washed with ethanol and the washings are boiled with NaOH and  $\text{H}_2\text{O}_2$ . By this means the parathion is hydrolysed to *p*-nitrophenol and the plant pigments are oxidised to colourless compounds. The *p*-nitrophenol is then determined absorptiometrically with the aid of a standard graph prepared from ethanolic solutions of parathion treated in the same way as the sample. To determine residues in the whole fruit, the macerated pulp is extracted with *n*-hexane, the *n*-hexane is removed and the residue is hydrolysed with alkali, the plant pigments being removed by oxidation with  $\text{H}_2\text{O}_2$  and subsequent extraction with ether after acidification. The *p*-nitrophenol is then extracted from the ethereal solution with NaOH and purified by re-solution in ether after acidification, and re-extraction from the ether with alkali. The optical density of the alkaline solution is then determined absorptiometrically and the parathion concn. is ascertained from a standard graph.

A. O. JONES

**2237. Stability of black mustard. I. Determination and stability of sinigrin in black mustard (*Sinapis nigra*).** O. Weis-Fogh (*Dansk Tidsskr. Farm.*, 1954, **28** [4], 69-83).—For the determination of sinigrin, a modification of the method of Meesemaker and Boivin (*cf. J. Pharm. Chim.*, 1930, **11**, 478) is preferred to the argentimetric method (*cf. Fr. Codex*, 1949, 7th Ed.). Free allyl isothiocyanate is first removed from the ground sample (5 g) by washing successively on a filter-paper with 20, 20 and 10 ml of light petroleum (boiling range 60° to 100° C). After macerating the dried sample in a tared 300-ml flask with 100 ml of water at 40° C for 30 to 120 min. (the time for complete enzymic hydrolysis of the sinigrin depends on the age of the sample after powdering), 100 ml of 10 per cent. aq.  $\text{NH}_3$  are added (in order to inactivate the enzymes) and allowed to act for 15 min. After cooling and making up the contents with water to 205 g, 20 g of 40 per cent. aq.  $\text{MgSO}_4$  are added, and the mixture is shaken and filtered. The sinigrin is determined by allowing 10 ml of 0.1 *N* I to act on 110 g of the acidified filtrate for 15 min. in darkness, and then back-titrating with 0.05 *N*  $\text{Na}_2\text{S}_2\text{O}_3$  (1 ml of 0.1 *N* I = 0.004956 g of allyl isothiocyanate, or 0.01986 g of sinigrin). The blank value of the chemicals used must be determined. Storage experiments, based on sinigrin determinations show that black mustard should be stored as the whole drug, and that the powdered drug (not defatted) keeps satisfactory only at R.H. > 60 per cent. and for limited periods. Moulds can cause spoilage of the drug by hydrolysis of the sinigrin.

P. S. ARUP

**2238. Annatto for dairy products.** British Standards Institution (B.S. 2450:1954, 6 pp.).—The

standard has been formulated with the object of reducing to a minimum the variations in the commercially available colouring matter known as annatto. The dye, which shall not contain extraneous colouring matter, is supplied in two forms: (i) butter colour (oil soluble), (ii) cheese colour (sol. in alkali). The oil and the alkali are defined and each solution should remain clear when stored at 15° C. The colours are defined in terms of (i) the C.I.E. system, (ii) Lovibond Tintometer units or (iii) an inorganic matching solution.

G. B. THACKRAY

**2239. The detection of trichloroethylene in cottonseed oil.** G. A. Wiese and C. L. Jesina (*Drug Standards*, 1954, **22** [5-6], 105).—To 2 ml of 10 per cent. w/v NaOH, add 2 ml of pyridine (reagent grade) and heat in a water-bath at 90° C for 5 min. Remove the tube from the water-bath and immediately add 1 ml of the cottonseed oil. The pink colour that develops in the pyridine layer within 20 min. varies from deep pink (1:100,000) to faint pink (1:300,000) if trichloroethylene is present.

N. E.

**2240. Separation and quantitative determination of thiamine and thiamine phosphoric esters and their preparation in pure form.** D. Siliprandi and N. Siliprandi (*Biochem. Biophys. Acta*, 1954, **14** [1], 52-61).—None of the present methods, chemical or biological, allow the separate determination of thiamine and each of its 3 phosphoric esters. Methods of chromatographic and electrophoretic separation of thiamine and its esters in biological material are described. *Paper chromatography*—Ascending chromatograms were run on Munkell OB paper for 15 hr. with various solvents, *n*-propanol - water - *M* acetate buffer (pH 5) (60:20:15) being satisfactory. Separation of 50  $\mu\text{g}$  of a mixture of the four substances was effected, the spots being developed as u.v. fluorescent thiochromes by a mixture of 96 per cent. ethanol (2 vol.), 10 per cent. NaOH (1 vol.) and 2.5 per cent.  $\text{K}_3\text{Fe}(\text{CN})_6$  (0.05 vol.). For quant. estimations 10 to 20  $\mu\text{l}$  of soln. containing < 30  $\mu\text{g}$  of individual substance were used, the spots being detected in u.v. light without spraying with alkaline ferricyanide, eluted with water and determined spectrophotometrically at 270  $m\mu$ . The error is - 5 per cent. *Paper electrophoresis*—Munkell 20 paper was used with acetate buffer (pH 5.44,  $\mu$  = 0.05) at 3.5 mA for 6 to 7 hr. Development of spots in the first method. Thiamine and monophosphothiamine are cathodal, and di- and triphosphothiamine are anodal. No breakdown of the esters occurred and there was no tailing. From 0.01 to 0.02 ml of solution containing 10 to 100  $\mu\text{g}$  of substance was used, but larger amounts (100 to 2000  $\mu\text{g}$ ) could be run on wide sheets. The error is less than - 5 per cent. Ion-exchange chromatography with Amberlite IRC 50 by the gradient elution technique also gives complete separation.

G. W. CAMBRIDGE

See also Abstract 2218.

## Sanitation

**2241. The detection of typhoid bacteria in water.** H. Thiele and E. Brinkmann (*Z. Hyg. Infectkr.*, 1953, **137**, 374-398).—The authors describe an investigation to determine the most suitable method of detecting typhoid bacteria in water and to develop a reliable procedure that would also be suitable for the bacteriological examination of

sewage and polluted surface waters. As Wilson and Blair's bismuth sulphite medium is specially suitable as a selective medium, experiments were made with the five forms of this medium. Best results were obtained with Lovreco's modification containing more ferrous sulphate, one-quarter the amount of sodium sulphite, and 10 g of bismuth ammonio-citrate instead of 6 g as in the original medium. This medium strongly checked accompanying bacteria and permitted easy recognition of typhoid and paratyphoid bacteria. A modified medium of Czernozubow with ten times the original concn. of brilliant green was found suitable for use with the Seitz filter enrichment method. Various enrichment methods by filtration, precipitation and evaporation were examined. The method recommended is filtration through an ultra-filter with subsequent washing of the filter; the apparatus required and the procedure used for this method are described. The method is suitable for examination of well and surface waters; for examination of sewage, evaporation of 1 or 2 ml of sample on Wilson - Blair plates is recommended.

## WATER POLLUTION ABSTR.

**2242. A new medium for the detection of enterococci in water.** W. Litsky, W. L. Mallmann and C. W. Fifield (*Amer. J. Publ. Hlth.*, 1953, **43**, 873-879).—It has previously been found that a satisfactory enrichment medium for the presumptive test for streptococci is Rothe's azide dextrose broth, and further studies have now been made to find a suitable confirmatory medium. A medium containing 0.4 per cent. of Na azide gave good growth of enterococci in 48 hr., and inhibited the growth of Gram-negative bacteria, but permitted the growth of some spore-forming bacteria. Ethyl violet, which has a bacteriostatic action on sporulating Gram-positive bacteria, was therefore added to the medium, which as finally used contained (per litre) tryptose 20 g, dextrose 15 g, NaCl 5 g,  $K_2HPO_4$  2.7 g, Na azide 0.4 g, and ethyl violet 0.00012 g. Good results were obtained when this medium was tested in confirmatory tests on polluted river water and sewage in conjunction with presumptive tests on glucose azide broth.

## WATER POLLUTION ABSTR.

**2243. Scheme for analysis of industrial water.** J. H. Phillips and K. G. Stoffer (*Ind. Eng. Chem.*, 1954, **46** [5], 970-974).—The outline of a comprehensive scheme for the analysis of industrial water is suggested. Two main groups of sample are recognised, viz., one for constituents affected by contact with air and the other for constituents unaffected by air. The first group is sub-divided so that analyses can be made either on a flow sample or on separate samples. The second group is sub-divided so that analyses can be performed on separate portions of a single sample or on separate samples.

J. H. WATON

**2244. Significance and application of water analysis data.** R. C. Ulmer (*Ind. Eng. Chem.*, 1954, **46** [5], 974-978).—Problems likely to be encountered in a plant water cycle are discussed, and the various analyses to be conducted on the water at various points in the system are detailed.

J. H. WATON

**2245. Analysis of water-formed deposits.** F. U. Neat and A. A. Berk (*Ind. Eng. Chem.*, 1954, **46** [5], 961-970).—A master scheme is proposed for the analysis of water-formed deposits. The preparation of the sample is described, and methods are

given for estimating all the elements likely to be present.

J. H. WATON

**2246. Bacteriological explanation of rate of oxygen consumption in the B.O.D. test.** A. M. Buswell, H. F. Mueller and I. van Meter (*Sewage Ind. Wastes*, 1954, **26** [3], 276-285).—The consensus of current opinion is that the B.O.D. reaction does not follow the monomolecular law and consequently that it is not possible to calculate total pollution load from O uptake data. Definite proof of this view is now afforded, and it is shown that the rate of O uptake in the B.O.D. of  $NH_3$  is related directly to the multiplication of *Nitrosomonas* sp. and independent of substrate concn., and that the 5-day dilution B.O.D. in p.p.m. of O is a measure of the total O requirements of all the bacterial processes occurring under the specific conditions during that period. A micro method for the determination of total org. C in sewage is outlined. Wet oxidation is followed by catalytic dry combustion in a micro-furnace. High-vacuum line procedures are used for purifying and transferring the  $CO_2$  that is evolved to a calibrated chamber for manometric measurement (cf. van Slyke *et al.*, *J. Biol. Chem.*, 1940, **136**, 509). In exploratory experiments with propionic acid, a recovery of 96 per cent. of the total C in the sample was achieved.

J. M. JACOBS

**2247. Investigation of the continuous recording of fluoride concentration in water.** K. F. Knowlton (*J. New Engl. Wat. Wks. Ass.*, 1954, **68** [1], 16-38).—A description is given of tests made to decide the suitability of a conductivity difference recorder for recording the concentration of fluoride in a municipal water supply. Equipment consists of two small cells that can be fitted into a pipe-line of reasonably small diameter at certain distances apart, and the conductivity difference can be recorded, integrated and correlated with the chemical addition. Operating results are given.

A. WEBSTER

**2248. Estimation of total organic carbon in aqueous solutions.** A. Gertner and H. Iveković (*Z. anal. Chem.*, 1954, **142** [1], 36-40).—Total organic carbon in aqueous solutions, drinking water and sewage is determined by oxidation with  $K_2S_2O_8$ , by using  $Ag^+$  as catalyst and weighing the  $CO_2$  formed. Results, in comparison with those by the chromic acid method, are satisfactory. Materials of known composition, e.g., oxalic acid, urea, hippuric acid, cysteine, dextrose and acetanilide give 100 per cent. recoveries. To the sample (diluted to 200 ml if necessary) add 10 per cent.  $AgNO_3$  (10 ml), 33 per cent.  $H_2SO_4$  (50 ml) and 10 per cent.  $K_2S_2O_8$  (100 ml), heat to 80°C and when the brown colour disappears add more 10 per cent.  $K_2S_2O_8$  (100 ml). The  $CO_2$  so formed is absorbed in suitably guarded soda-lime tubes.

P. S. STROSS

**2249. Simplified method for determination of oil in waste waters.** R. E. Beaudoin (*Sewage Ind. Wastes*, 1954, **26** [4], 568-569).—Large samples (1-gal.) of industrial wastes consisting of oil, water, dirt and debris, which are received from metal working plants, are best handled by siphoning off part of the water, adding a weighed amount of solvent to the contents of the sample bottle and setting the stoppered bottle aside until the supernatant layer of solvent with the oil dissolved in it becomes free of the dirt, which settles to the bottom of the layer. The oil content of an aliquot of this solution is then determined.

J. M. JACOBS

**2250. Determination of aldrin and dieldrin in formulations by partition chromatography.** H. F. Beckman (*Anal. Chem.*, 1954, **26** [5], 922-923).

Aldrin (an insecticide containing > 95 per cent. 1:2:3:4:10:10-hexachloro-1:4:4a:5:8:8a-hexahydro-1:4:5:8-dimethanonaphthalene) and dieldrin (> 85 per cent. of 1:2:3:4:10:10-hexachloro-6:7-epoxy-1:4:4a:5:6:7:8:8a-octahydro-1:4:5:8-dimethanonaphthalene) are determined chromatographically on silicic acid with *n*-hexane saturated with nitromethane as the mobile solvent. The procedure may be applied to the determination of aldrin or dieldrin, singly, and in combination with DDT and to aldrin in combination with S. Recoveries of aldrin, dieldrin and DDT are > 97 per cent. G. P. COOK

**2251. Organo-phosphorus insecticides. Biological assay of phosphorus insecticides.** J. F. Newman (*Chem. & Ind.*, 1954, [22], 617-619).—This is a review of biological methods of assay for organic phosphorus insecticides. Chemical methods are often too insensitive, unspecific or not available. Biological methods follow two main types—the application of graded doses to groups of animals and measuring the percentages affected, and the measurement of the inhibition of the enzyme cholinesterase *in vitro*. Methods are described involving the adult flies of *Musca domestica* (housefly) or *Drosophila spp.* (fruit fly), the crustacean *Gammarus pulex* and *Daphnia pulex* and the larvae of the mosquito *Aedes aegypti*, which are particularly sensitive. The assay of residues from plant materials requires a preliminary extraction to remove interfering toxic substances. It is emphasised that bio-assay methods do not provide reliable data on the toxicity of insecticides in man. G. F. SOMERS

**2252. Qualitative and quantitative estimation of E605 in biological materials.** E. Pfeil and H. Goldbach (*Klin. Wochschr.*, 1953, **31** [41-42], 1011-1012).—A method has been developed for estimation of E605 (Parathion or O:O-diethyl O-*p*-nitrophenyl thiophosphate) based upon splitting off *p*-nitrophenol by treatment with alkali and subsequent identification of this compound by paper electrophoresis. Preliminary tests indicated that heating with alkali is necessary since E605 appears to be more resistant to alkali when fixed in tissues. Homogenised material is treated with ethanol and tartaric acid, and after several hours is filtered; the filtrate is treated with alkali and the alcohol is evaporated on a water-bath. The residue is acidified with  $H_2SO_4$  and extracted with ether, the ether is evaporated and the residue is treated with  $NH_3$  and subjected to ascending paper chromatography in ethanol- $NH_3$ -water (80:4:16).  $R_F$  values are: *p*-nitrophenol, 0.71, *o*-nitrophenol, 0.69, dinitrophenol, 0.74, Naphthol yellow S, 0.45. *p*-Nitrophenol separated with butanol-pentanol- $NH_3$  has an  $R_F$  value of 0.28. After chromatography, the fractions can be eluted from the paper and their extinctions can be determined by u.v. spectrophotometry. The quant. determination of *p*-nitrophenol will thus be equivalent to the E605 present in the original sample. G. W. CAMBRIDGE

**2253. Cockroach spray method. Official method of the Chemical Specialties Manufacturers' Association for evaluating cockroach sprays.** Anon. (*Soap, N.Y.*, 1954, Blue Book, 249-250).—The method described is a modified composite form of the Bottimer and Hazard methods of testing cockroach sprays. Details are given of (i) apparatus, including

reference insecticide, test insect, rearing room, testing room, spray chamber, atomiser, treatment container and recovery dishes; (ii) procedure, including rearing of test insects, test procedure and assembly and evaluation of data; and (iii) conditions for evaluation with a tabulated example of test data. G. HELMS

**2254. The colorimetric determination of benzene hexachloride in insect tissues.** F. R. Bradbury and H. Standen (*J. Sci. Food Agric.*, 1954, **5** [5], 252-256).—In the colorimetric determination of benzene hexachloride involving hydrolysis, nitration and hydrolysis again (Armstrong, Bradbury and Standen, *Ann. Appl. Biol.*, 1951, **38**, 555) the main colour-producing component is 6-chloro-2:4-dinitroresorcinol; 2-chloro-4:6-dinitro- and 5-chloro-2:4-dinitroresorcinol may also contribute to the colour. An improved method for extracting the insecticide from insect tissue is described: the insects are finely ground with  $Na_2CO_3$  (anhydrous) and extracted 3 times with a total of 20 ml of  $CCl_4$ . Fats, etc., are removed from the combined extracts by passing them through a column (2 cm diam. and 10 cm long) packed by adding a stiff slurry of 3 g of high-grade kieselguhr (Kensil P11) with 2 ml of 30 per cent. oleum and sufficient  $CCl_4$  and allowing to drain. The column is washed 4 times with 3 to 4 ml of  $CCl_4$  and the determination is carried out on the combined eluates, beginning with the first nitration. S. C. JOLLY

**2255. Black and white disinfectant fluids.** British Standards Institution (B.S. 2462:1954, 11 pp.).—General requirements are specified for disinfectant fluids of coal-tar type covering 5 groups of black and 6 groups of white fluids. The fluids are grouped according to their germicidal value as determined by a specified method of test (Rideal-Walker, Chick-Martin, or "Crown Agents," a min. value only being specified for each group. The technique of the Crown Agents' test is described in an appendix. The general requirements include standards of stability before and after dilution, odour, corrosive action, and packaging. H. F. W. KIRKPATRICK

See also Abstract 2236.

### Agriculture and Plant Biochemistry

**2256. The determination of boron in boron-containing mineral fertilisers by the ester method.** H. Roth and W. Beck (*Z. anal. Chem.*, 1954, **141** [6], 404-414).—Because of interfering ions, B cannot be directly estimated, but is first quant. distilled as the methyl ester. This is hydrolysed and estimated volumetrically or colorimetrically. To mineral fertiliser containing 0.1 per cent. or more of B (1 to 2 g), add an equal weight of  $Ca(OH)_2$  and heat to 580°C for 30 min. to remove nitrate. Transfer to a distillation flask, using only 4 ml of  $H_2O$  and 45 ml of methanol and add, while shaking, conc.  $H_2SO_4$  (4 ml); temp. must not exceed 50°C. Blow methanol vapour into the flask, which is fitted with a vertical air reflux condenser and a water condenser, keeping the temp. below 75°C. Collect the alcohol-ester mixture in 0.5 N NaOH (10 ml), evaporate the methanol and, after neutralising the excess of NaOH and boiling to remove  $CO_2$ , add mannitol and titrate the boric acid with 0.02 N NaOH (bromocresol purple). Alternatively a colorimetric estimation with anthrimide (1:1'-dianthraquinonylamine) may be used. The ester is then collected in  $Ca(OH)_2$  and evaporated

to dryness. To the dry residue, add conc.  $H_2SO_4$  (2 ml), anthrimide reagent (5 ml) (100 mg of anthrimide in 500 ml conc.  $H_2SO_4$ ) and heat to  $90^\circ C$  for 3 hr. Make up to 10 ml with conc.  $H_2SO_4$  and measure the absorption through a S61 filter. As little as  $1 \mu g$  can be assayed.

P. S. STROSS

**2257. The assay of boron in plants and soils by the ester method.** H. Roth and W. Beck (*Z. anal. Chem.*, 1954, **141** [6], 414-418).—In order to carry out the assay for B (see abstract 2256) plants may be dried at  $80^\circ C$ . The B content of the different parts of a plant is given; most boron is found in growing tissues. Analysis of B in soil is carried out in the same way. Up to 10 ml of water may be added to the original sample provided that the quantity of  $H_2SO_4$  is increased to 20 ml.

P. S. STROSS

**2258. The polarographic estimation of molybdenum in plant materials.** G. B. Jones (*Anal. Chim. Acta*, 1954, **10** [6], 584-590).—The catalytic wave of Mo in a soln. of  $H_2SO_4$  and  $NaClO_4$  (cf. Haight, *Brit. Abstr. C*, 1952, 95) is used for its polarographic estimation.  $\alpha$ -Benzoinoxime is used to separate the Mo from other metals (Cu and Bi) that would interfere. The lowest concn. that can be measured is  $0.02 \mu g$  of Mo per ml, when the error may be  $\pm 20$  per cent.; at a concn. of  $0.1 \text{ mg per ml}$  the error may be  $\pm 5$  per cent. **Procedure**—For plant materials containing  $> 1 \text{ p.p.m.}$  of Mo, digest 1 g of the dried sample with  $H_2SO_4$ ,  $HNO_3$  and  $HClO_4$ . Dilute with water to produce a 1 to 2 per cent. concn. of  $H_2SO_4$ , add 2 per cent. ethanolic  $\alpha$ -benzoinoxime (2 ml) and extract with  $CHCl_3$ . Evaporate the  $CHCl_3$  from the extracts, treat the residue with  $H_2SO_4$  (7 drops), decompose the organic matter by heating with 2 to 3 portions (10 drops) of  $HNO_3$  and finally with  $HClO_4$  (2 drops). Evaporate to fumes of  $H_2SO_4$ , cool, add  $M NaClO_4$  (4.8 ml) and polarograph at  $-0.1$  to  $-0.8 \text{ V}$  vs. the anode pool. The  $E_{1/2}$  value for Mo is  $-0.56 \text{ V}$ .

W. C. JOHNSON

**2259. Identification and determination of non-nitrogenous organic acids of sugar cane by partition chromatography.** E. J. Roberts and L. F. Martin (*Anal. Chem.*, 1954, **26** [5], 815-818).—Application of partition chromatography, mainly by use of the procedures of Marvel and Rands (*J. Amer. Chem. Soc.*, 1950, **72**, 2642), has effected the separation of nine non-nitrogenous acids from sugar-cane juice. Separation of the acids on the large-capacity silica gel columns has made it possible to obtain the acids from small quantities of juice in amounts sufficient for purification and complete identification. The method gives more satisfactory confirmation of the presence of malic, oxalic, citric and glycolic acids, for which there was previous evidence, and the hitherto undetected acids, which include mesaconic, fumaric and succinic. The technique of applying an acidified, pre-adsorbed soln. of the total lyophilised solids directly to the columns avoids the necessity for preliminary separations or concn., through which some of the acids may be lost. The recoveries of all the organic acids were found to be essentially quant., except that of oxalic acid, which was erratic.

G. P. COOK

**2260. Improved chromatographic method for analysis of sugar beet products.** R. F. Serro and R. J. Brown (*Anal. Chem.*, 1954, **26** [5], 890-892).—The determination of raffinose, meso-inositol and

galactinol in process liquors is described. Raffinose is determined by dilution of the sample to a suitable concn., and after a chromatogram has been run in a solvent consisting of *n*-propanol, ethyl acetate and water (7:1:2 by vol.), the raffinose spots are developed with a 1-naphthol soln. Galactinol and inositol are determined on a separate chromatogram, which is run with two solvents. The first solvent (15 vol. of benzyl alcohol, 5 vol. of *tert*-butanol and 1 vol. of water, to which is added 1 per cent. by vol. of 90 per cent. formic acid) removes interfering constituents of the juice and the second solvent (10 vol. of water-saturated 2:4:6-collidine and 1 vol. of the collidine) separates the carbohydrate constituents and permits quant. determination of the galactinol and inositol. These spots are developed with ammoniacal  $AgNO_3$ .

G. P. COOK

**2261. Determination of raffinose in beets by the chromatographic method.** M. M. Chollet (*Ind. Agric. et. Alim.*, 1954, **71**, 205-208).—Samples of pressed beet juice, defecated and concentrated, were chromatographed, with ethyl acetate-acetic acid-water (9:3:4) as solvent by a procedure based on that of Blass *et al.* (*Bull. Soc. Chim. Biol.*, 1950, **32**, 130). Results for two varieties of beets and of some analyses of vinasses are tabulated. There is a considerable loss in raffinose in the processing of the beets. This may be due to combination of raffinose with melibiose formed by hydrolysis, or with nitrogen compounds, the products being removed in defecation.

SUGAR IND. ABSTR.

**2262. The quantitative determination of choline in plant material in the presence of lecithin.** L. Acker and G. Ernst (*Z. anal. Chem.*, 1954, **142** [1], 5-12).—Remove lecithin from the soln. containing a mixture of it and choline by extracting five times (shaking 15 min.) with ether; emulsions are broken by adding methanol and  $(NH_4)_2SO_4$ . Remove ether from the aqueous phase by heating, add sufficient  $HNO_3$  to make the concn. 20 per cent. and reflux for 2 hr. After cooling neutralise the soln. with 33 per cent. NaOH. To an aliquot (1 to 2 ml) in a conical centrifuge tube, add 0.3 ml of iodine solution (157 g of I and 200 g of KI to 1 litre), allow to stand for 10 min. at  $0^\circ C$  and centrifuge. Rapidly filter the supernatant liquid through a sintered-glass filter and wash the centrifuge tube and filter 3 times with 1 ml of saturated NaCl soln at  $0^\circ C$ . Dissolve the yellow needles in the centrifuge tube and on the filter in chloroform, add starch, and titrate immediately with 0.01 *N*  $Na_2S_2O_3$ . 1 ml of 0.01 *N*  $Na_2S_2O_3 \equiv 0.1335 \text{ mg}$  of choline.

P. S. STROSS

**2263. The isolation of L-citramalic acid from the peel of the apple fruit.** A. C. Hulme (*Biochim. Biophys. Acta*, 1954, **14** [1], 36-43).—Detection of an acid ("G" acid) differing in behaviour from any of the known fruit acids has been previously reported (Hulme, *Nature*, 1953, **172**, 344); it was suggested that this might be  $\beta$ -hydroxyglutaric acid or the isomeric citramalic acid ( $\alpha$ -methylmalic acid). A method of extraction of this acid from the peel of Edward VII apples and its paper-chromatographic and chemical characterisation are reported. An ethanol extract of frozen peel is freed from alcohol by evaporation, treated with charcoal, the cations and amino-acids are removed by Zeokarb 215 and the organic acids are collected

on a column of De-acidite E (Hulme, *J. Exp. Bot.*, 1951, 2, 296). Displacement with 0.1 *N* HCl produced fractions containing malic acid and further acids including the "G" acid under investigation. Complete separation from malic acid with Dowex 2 could not be achieved, but "G" acid was found to be more soluble in isobutyl methyl ketone than malic acid, and could be separated on phosphate-buffered silica gel (pH 2.9). Partition chromatography resulted in fractions containing only "G" acid, which was crystallised. It was characterised by chemical methods, by comparison with  $\beta$ -hydroxyglutaric acid and DL-citramalic acid, by paper chromatography and by its i.r. absorption spectrum. Titration curves of citramalic,  $\beta$ -hydroxyglutaric and malic acid were compared. It is concluded that "G" acid is L-citramalic acid. The addition of a  $\text{CH}_3$  group to malic acid produces an identical shift in the  $R_F$  value in one acid solvent (benzyl alcohol - *tert*-butanol - isopropanol - formic acid - water) whereas another acid solvent (*n*-butanol - formic acid - water) shows up the structural difference of the two isomeric dicarboxylic hydroxy acids. G. W. CAMBRIDGE

**2264. A new method of absorption analysis for estimating carotene in the pigments of green leaves.** R. Köppen (*Kolloidztschr.*, 1954, 135 [3], 150-159).—The use of various adsorbents for the chromatographic separation of carotene from extracts of green leaves is investigated. The more active forms of alumina retain some of the carotene. A simplified method of separation is developed in which 10 ml of extract are shaken with 0.5 g of  $2\text{SiO}_2 \cdot \text{H}_2\text{O}$ . After centrifugation or filtration, the carotene, which remains unadsorbed in the liquid, can be estimated photometrically with the aid of a violet filter. The removal of other pigments can be checked by measuring the approx. absorption spectrum with a series of 5 filters. A. B. DENSHAM

**2265. Determination of residual Crag herbicide 1 and its hydrolysis products on food crops.** J. N. Hogsett and G. L. Funk (*Anal. Chem.*, 1954, 26 [5], 849-853).—Crag herbicide 1 [ $\text{Na } 2-(2:4\text{-dichlorophenoxy})\text{ethyl sulphate}$ ] is determined by measurement of the intensity of the coloured complex it forms with methylene blue chloride. An aliquot of the aq. extract of the plant material is added to an aq. soln. of methylene blue chloride and the complex formed is extracted into  $\text{CHCl}_3$ . The absorbance of this solution is measured at 650  $\text{m}\mu$  and the concn. of herbicide is found from a calibration curve. Hydrolysis products are extracted into  $\text{CHCl}_3$ , sulphated by treatment with chlorosulphonic acid, extracted into water and determined essentially as for the parent compound. The recovery from synthetic samples is generally  $> 92$  per cent., with an average standard deviation of  $\pm 14$  per cent. G. P. COOK

## 5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

### General

**2266. Micro-beakers.** British Standards Institution (B.S. 1428: Part E 2:1954, 6 pp.).—The general shape and dimensions are specified of the following types of glass and silica micro-beakers: tall, of nominal capacity, 1, 5, 7, 15 and 20 ml;

squat, 3 and 10 ml; and conical, 5, 10 and 20 ml. Covers are also specified, 22, 27 and 34 mm in diameter. H. P. PAGET

**2267. Soxhlet extractors.** British Standards Institution (B.S. 2071:1954, 11 pp.).—Specifications are given for two types of extractor for general laboratory use, together with recommendations on the most suitable types and sizes of condensers, flasks and thimbles for use with them. Type 1 has the siphon tube bent in a loop, while type 2 has a concentric siphon tube. The bore of the siphon tube was chosen so as to give satisfactory refluxing with a variety of solvents. The extractors are of four sizes: 40, 60, 100 and 200-ml nominal capacities. H. P. PAGET

**2268. Apparatus for hot filtration, extraction and recrystallisation with volatile and [in]flammable solvents.** H. Shiba (*Anal. Chem.*, 1954, 26 [5], 943).—A description and drawing of a simple all-glass hot filter whereby recrystallisation with volatile solvents can be effected easily without loss of solvent or deposition of crystals on the filter-paper. The solvent-containing conical flask is connected to a specially constructed filter funnel, the constricted upper rim of which fits into the belled-out lower end of the condenser. All joints are ground-glass. The apparatus can also be used for continuous extraction of solids. W. J. BAKER

**2269. A melting-point apparatus of bath type with motor-driven stirrer.** C. W. Ballard, P. G. W. Scott and J. A. Simpson (*J. Pharm. Pharmacol.*, 1954, 6 [6], 430-432).—An apparatus for determining the melting points of 3 specimens simultaneously, consisting of an upright oil-bath heated by a Meker burner and stirred by a motor-driven Archimedean screw housed in a vertical tube, is described and illustrated. C. E. SEARLE

**2270. Proposed specifications for A.S.T.M. hydrometers.** Anon. (*Proc. A.S.T.M.*, 1952, 52, 527-532).—The specifications cover constant-mass variable-displacement hydrometers made of glass. The stem must be uniform in cross-section and extend  $\leq 15$  mm above the top graduation and  $\leq 3$  mm below the lowest graduation. The paper scale must be anchored by the design of the stem or fixed by a cement that does not soften below 105° C. When tests are made at three scale points these must include  $\leq 60$  per cent. of the graduated interval of the scale, and neither of the extreme points must be farther from the nearest end of the graduated scale than 25 per cent. of its length. J. M. JACOBS

**2271. Cryoscopic method of determination of molecular weights by means of resistance thermometers.** V. Ya Mikhelson (*J. Anal. Chem., U.S.S.R.*, 1954, 9 [1], 22-28).—The special resistance thermometer described is made of semi-conducting material and capable of measuring temperature differences to an accuracy of 0.001°; it is superior to a Beckman thermometer for mol. wt. determinations. Accurate determinations can be made with 5 to 20 mg of substance in 7 ml of solvent with the apparatus described and a determination can be carried out in 30 to 40 min. G. S. SMITH

**2272. A simple osmometer for the rapid determination of molecular weights.** M. F. Mallette (*Arch. Biochem. Biophys.*, 1954, 48 [2], 315-321).—Details of the construction and use of a simple

collodion-membrane osmometer are given. The apparatus requires 2 to 3 ml of test soln. and is suitable for mol. wt. determinations in the range 20,000 to 70,000. W. H. C. SHAW

**2273. Automatic recording of ion concentration in flowing solution.** J. A. Lewis and K. C. Overton (*Analyst*, 1954, **79**, 293-297).—An apparatus is described that automatically and continuously records the concn. in flowing or stationary solutions of metallic ions that give discrete, well-formed polarographic waves. It consists essentially of two polarographs in opposition. Results with a test solution of  $\text{BiCl}_3$  and  $\text{CdCl}_2$  in dil. HCl, in which the concn. was varied (0 to 500  $\mu\text{g}$  of the metals per ml), showed that the instrument followed the change in the Cd concn. The readings of the continuous recorder can be used to indicate variations with time of the concn. of a metal that was being used up, e.g., in a plating bath. A. O. JONES

**2274. An improved apparatus for the micro-determination of unsaturation in organic compounds by catalytic hydrogenation.** A. F. Colson (*Analyst*, 1954, **79**, 298-303).—The apparatus described and illustrated is an improved form of that of Johns and Seiferle (*Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 841); it can be used for hydrogenation of solid or volatile liquid samples at temp. ranging from 0° to 50° C. The manipulation of the apparatus is described in detail, and results (as mg of sample absorbing 1 millimol. of H) are given for sorbic acid, cinnamic acid and cyclohexene. The catalyst used is Adam's platinum oxide. The relative error in routine determinations is  $\approx \pm 2$  per cent. A. O. JONES

**2275. High-frequency titration.** V. A. Zarinsky and D. I. Koshkin (*J. Anal. Chem., U.S.S.R.*, 1954, **9** [1], 29-36).—Apparatus for high-frequency titration with a 4-megahertz generator stabilised by a quartz crystal is described. It is applied to acid-alkali, oxidation-reduction and precipitation titrations. G. S. SMITH

**2276 All-glass atomiser for chromatographic analysis procedures.** V. H. Ortegren (*Anal. Chem.*, 1954, **26** [5], 943-944).—The device described and illustrated provides easily-controlled positive atomisation and ensures that larger droplets of liquid are automatically returned to the container. Liquid is supplied to the atomiser through a rigidly-supported capillary tube, flow being caused by creation of low pressure by the high-velocity air from the atomising nozzle. The air-conduit is also connected to a short diversion tube ending in a finger-controlled valve, the operation of which controls the atomisation. The apparatus, which can be made in the laboratory, can also be used in an open vessel or test tube. W. J. BAKER

**2277 Automatic receiver changer [for chromatographic analysis].** E. S. Sanderson (*Anal. Chem.*, 1954, **26** [5], 944).—The mechanical receiver changer described and illustrated is actuated by the wt. of the sample collected and operates continuously, with slight attention, for  $< 120$  hr., during which 500 to 600 samples (5 to 7 ml) of effluent can be collected in test-tubes (150 by 16 mm). The apparatus, which is easily made in the workshop, is very suitable for use with chromatographic columns involving separation of several grams of a complex mixture. W. J. BAKER

**2278 Precise measurements with Bingham viscometers and Cannon master viscometers.** J. F. Swindells, R. C. Hardy and R. L. Cottingham (*J. Res. Nat. Bur. Stand.*, 1954, **52** [3], 105-120).—A critical study is made of the techniques used at the National Bureau of Standards with Bingham and Cannon viscometers, and the corrections applicable are discussed. Instruments of each type were calibrated with  $\text{H}_2\text{O}$  at 20° C and used to determine  $\eta$  for four hydrocarbons in the range 0.4 to 40 centipoises. For each liquid the results agree to within 0.05 per cent., indicating no gross error with either instrument. The Cannon viscometer is preferred, because of its inherent relative simplicity of operation; re-design of the fiducial bulb should simplify the head correction for the difference in surface tension of  $\text{H}_2\text{O}$  and oil. A. B. DENSHAM

**2279. Gas scrubbing towers.** B. J. Heywood (*M. & B. Lab. Bull.*, 1954, **1** [1], 6).—An arrangement is described and illustrated in which waste water from a condenser flows down through a column packed with Raschig rings and washes or absorbs counterflowing gases such as  $\text{SO}_2$ , HCl, HBr or Cl, which are evolved in many laboratory-scale organic reactions. The water flows up through an annular space surrounding the column and is sprayed on to the top of the rings. Revolatilisation of less-soluble gases such as Cl can be prevented by allowing NaOH soln. to drip on to the top of the column or by introducing  $\text{SO}_2$  through a gas inlet. B. J. W.

**2280. Improved automatic apparatus for pharmacological assays on isolated preparations.** A. Boura, J. L. Mongar and H. O. Schild (*Brit. J. Pharmacol.*, 1954, **9** [1], 24-30).—An apparatus for pharmacological assay on isolated preparations, where the organ bath is emptied and filled automatically, has previously been described by Schild (*Brit. J. Pharmacol.*, 1946, **1**, 135; 1947, **2**, 189). In that apparatus, the drug had to be added by hand and the stages in the assay cycle were of fixed duration. In the proposed modification of this apparatus, all stages, including the addition and removal of drug and the control of the drum, are automatic. The various stages in the cycle are independently variable, the drugs can be given in any order and an antagonistic or potentiating drug can be added at some stage in the cycle. The apparatus is further modified so that the solutions do not come into contact with rubber tubing. G. B. CHESHER

**2281. Trials with an experimental long-period sampler of airborne dust.** J. G. Dawes, S. R. Howarth and A. Slack (*Minist. Fuel. Pur. Safety Min. Res.*, 1954, *Res. Rep. No. 87*, 9 pp.).—An apparatus is described for continuous sampling of dust in mines throughout the period of a shift. Air is drawn upwards through filter-paper by a constant level water aspirator at 1.25 ml per min. in such a way that particles  $> 6 \mu$  are not collected. Methods of determining the density of the deposit by an optical densitometer or by the increase in resistance to flow are described. A. B. DENSHAM

**2282. Proposed method of sampling steam and water at sub-atmospheric pressure.** Anon. (*Proc. A.S.T.M.*, 1952, **52**, 503-504).—The procedure and apparatus are as described in A.S.T.M. designations D 1066 and D 1192, together with means for extracting a sample at sub-atm. pressure and

delivering it to a container at atm. pressure, e.g., an atm. leg, a small (diaphragm-type) condensate pump, controlled by the level in a collecting chamber, vac. pumps, etc. A flow (at a rate of  $< 50$  lb per hr.) is established through the sampling system and the condensed or cooled effluent is discarded until the lines and cooling system are completely flushed out, as determined by conductivity or other measurement. J. M. JACOBS

**2283. Chemical instrumentation in the pulp and paper industry.** J. Grant (*Fibres*, 1954, 15 [4], 119-122, 130).—Digestion in order to separate non-cellulosic constituents may be controlled by measuring the electrical conductivity of the digesting fluid; the consumption of the ionic reagent, e.g., NaOH, causes a fall in the conductivity. In bleaching, temp., consistency of the wet pulp suspension and pH should all be regulated. Beating may be controlled by means of the "Canadian freeness tester" and in the next process, sizing, pH control is again important. Instruments for measurement of the moisture content of paper, control of drying conditions, and for determining the bursting, tensile folding, and tearing strengths of paper are also discussed. L. VALENTINE

**2284. Testing and measuring instruments devised in the Wool Industries Research Association laboratories.** Anon. (*Fibres*, 1954, 15 [4], 123-125).—Descriptions are given of the following instruments: projection microscope, fibre-fineness meter, fibre-length machine, roving levelness tester, yarn crimp meter, water-repellency tester and a fabric abrasion machine. L. VALENTINE

See also Abstracts 2090, 2247.

### Optical

**2285. Aluminium powder as a binder in sample preparation for X-ray spectrometry.** I. Adler and J. M. Axelrod (*Anal. Chem.*, 1954, 26 [5], 931-932).—The sample or internal standard (0.5 g of minus 200-mesh material) is intimately mixed (under ether) with 0.5 g of Al dust (minus 270-mesh), and the mixture is then compressed into a strong disc of 1 in. diam. and 0.05 in. thick. Loss of abs. intensity due to dilution by Al powder is  $\approx 5$  to 10 per cent., and the max. variation in homogeneity of the briquettes is 1.3 per cent. The method is very satisfactory for the X-ray fluorescence spectrometric analysis of ores and minerals. W. J. BAKER

**2286. The construction of a combined sources unit for emission spectrography.** R. G. Stone and H. L. Bolton (*J. Sci. Instrum.*, 1954, 31 [5], 175-178).—A unit is described that includes the circuits for two intermittent a.c. arcs, one pulsed d.c. arc, one constant current d.c. arc, a condensed spark and a conventional d.c. arc operated from mains supply. G. SKIRROW

**2287. A high-precision slit and photo-cell arrangement for spectrochemical analysis.** C. G. Carlsson (*Spectrochim. Acta*, 1954, 6 [3], 211-215).—In the arrangement described and illustrated the position along the focal plane of each of the four exit-slits (width  $20 \mu$ ) is adjusted from the outside of the spectrograph to within 1 to  $2 \mu$ , whilst each slit is adjusted separately to coincide with the focal plane and be parallel to the spectrum line. By fixing a strip of fine-grain photographic film on the steel edges of the slit, full information can be obtained about the accuracy of alignment at a

point within a film-thickness of the slit. Good resolution of the P (2149-11 Å) and Cu (2148-97 Å) lines from those of Fe in the spectra of P-Cu steels is thus possible. W. J. BAKER

**2288. An electronic timing circuit for spectroscopic analysis.** W. Seith and H. de Laffolie (*Spectrochim. Acta*, 1954, 6 [3], 216-222).—The circuit described and illustrated ensures automatic control of all normal operations in the recording of spectra. It also controls the interrupted arc. Pre-sparking and exposure times can be adjusted continuously from 2 sec. to 8 min., whilst exposure times are reproducible within 0.3 per cent. W. J. BAKER

**2289. A new sample-stand for spectrochemical analysis.** W. Koehler (*Spectrochim. Acta*, 1954, 6 [3], 223-227).—The sample-stand is fitted with a binocular microscope for observing the counter-electrode and the sample-spot; hence the spark may be located and its progress followed. The diameter of the sparked area can be restricted to 0.2 mm for a 2-min. exposure. The arrangement can be adapted to the spectroscopic examination of inclusions (e.g., in silver-plate), soldered zones and intermetallic layers. W. J. BAKER

**2290. Controllable interference-type optical filter.** R. Blythe (*J. Opt. Soc. Amer.*, 1954, 44 [4], 336-339).—A transmission-type interference filter has partially aluminised quartz flats as optical elements, one of which is supported on a nickel tube that passes through a short solenoid. This tube shortens slightly in accordance with the strength of the magnetic field produced by the solenoid; hence the separation between the two quartz plates can be varied according to the magnitude of a d.c. electrical signal. An increase of current from zero to 400 mA shifts the transmission band, which for the coatings used is about  $110 m\mu$  wide, from the violet through the visible spectrum to the red. B. S. COOPER

**2291. Note on the polishing of rock-salt windows.** E. J. Slowinski, jun. (*J. Opt. Soc. Amer.*, 1954, 44 [4], 342).—After cutting and rough grinding with No. 280 abrasive, the window is polished on a carefully prepared pitch lap by means of a saturated salt solution as abrasive. By this method, windows up to 40 mm square may be produced scratch-free, clean, and plane to within a few interference fringes. B. S. COOPER

**2292. Some interferences in flame photometry.** R. D. Caton, jun., and R. W. Bremner (*Anal. Chem.*, 1954, 26 [5], 805-813).—The effects on flame intensity of variations in viscosity and spray particle size caused by addition of org. solutes during the flame-spectrophotometric determination of Na, K and Ca have been examined for concn. up to  $\approx 4 M$  of sucrose, glucose, urea and gelatin. None of these additives produces any measurable background illumination, but flame intensity is decreased with increasing concn. of additive, e.g., a 2 per cent. decrease is produced by sucrose and gelatin in concn. of 2 to 3 and 0.1 to 0.2 mg per ml, respectively. The decrease is not caused by viscosity alone, and probably arises from the effects of the additive on the spray characteristics, with consequent hindering of activation of the metal by the flame. Methods of correcting for interferences of org. contaminants are discussed briefly, viz., by dilution of the sample or by the compounding of standards. W. J. BAKER

**2293. Absorption spectrum sorter.** T. Elder and W. Benesch (*J. Opt. Soc. Amer.*, 1954, **44** [4], 279-283).—A modification to a conventional double-beam infra-red spectrometer provides a record in which the absorption spectra of two substances appear on either side of a central base line. The applications of this principle to the handling of comparison spectra, to the identification of absorbing constituents and to the enhancement of resolution are discussed. B. S. COOPER

**2294. Water-prism ultra-violet monochromators.** D. J. Fluke and R. B. Setlow (*J. Opt. Soc. Amer.*, 1954, **44** [4], 327-330).—Two large-aperture monochromators are described that are suitable for obtaining relatively intense monochromatic u.v. radiation in the 300 to 200-m $\mu$  range. The first instrument has a dispersing system formed by immersing a concave mirror 8 in. in diam. in water, so that the free water surface and the mirror form approximately a 30° prism. The second instrument, of more conventional arrangement, has a 60° water prism enclosed by quartz plates having 10 in. diam. apertures. The second instrument has improved resolving power and can resolve the 2400 Å and 2375 Å Hg lines; at 240 m $\mu$  the stray radiation amounts to about 5 per cent. The instruments provide monochromatic radiation of sufficient intensity for use in biological experiments. B. S. COOPER

**2295. Continuous-flow cell for absorption measurements on solutions which fade.** G. P. Cooke (*J. Sci. Instrum.*, 1954, **31** [5], 180-181).—The cell described reduces errors in absorption measurements on solutions that fade under the action of the incident radiation, by allowing a quantity of the solution to flow continuously during the measurements.

G. SKIRROW

**2296. Note on the infra-red spectra of powders.** R. Lejeune and G. Duyckaerts (*Spectrochim. Acta*, 1954, **6** [3], 194-197).—Quant. i.r. spectroscopy of mg amounts of solids (insol. in usual solvents) by incorporation of the finely-ground solid ( $\approx 5$  mg) in a sintered disc (25 by 0.7 mm) of KBr as internal standard is examined experimentally. Data for calcite of particle-diam. ( $d$ ) from 2 to 40  $\mu$  show that the absorption intensity of the bands (11.4 and 14.02  $\mu$ ) depends very largely on  $d$ , max. and const. absorption occurring only at small values of  $d$ . Observed values of the mean coeff. of absorption agree with those calculated from the theoretical equation relating all the parameters involved.

W. J. BAKER

**2297. Continuous measurement of atmospheric ozone by an automatic photo-electric method.** R. Stair, T. C. Bagg and R. G. Johnston (*J. Res. Nat. Bur. Stand.*, 1954, **52** [3], 133-139).—The automatic photo-electric recorder described continuously measures atmospheric ozone at night, the Hartley band at 250 to 260 m $\mu$  being used. The source is a low-pressure mercury arc at 1450 ft from the IP28 photomultiplier used as detector. The light beam is modulated at 510 cycles per sec., and the amplified output is fed to a recorder. Continuous absorption by smoke, etc., is corrected for by automatic insertion in turn of filters passing bands centred at 253.7, 365.5 and 405 m $\mu$ . Measurements at Washington show increases from a normal concn. of a few parts per 1000 million to several parts per 100 million associated with the passage of a cold front.

A. B. DENSHAM

**2298. A colorimeter for pyrotechnic smokes.** I. Nimeroff and S. W. Wilson (*J. Opt. Soc. Amer.*, 1954, **44** [4], 299-302).—The photo-electric colorimeter described has been designed to measure the chromaticity co-ordinates of coloured smokes. The sample chamber, a 3-ft cube, is illuminated through one face (plate glass) by four 150-W tungsten-filament lamps. A set of tri-stimulus filters has been devised for use with RCA type 5891 multiplier photo-cells, so that the results obtained from the instrument are in terms of daylight illumination (6500° K), although the instrumental illuminant (tungsten filament) is at a much lower colour temperature (2780° K). B. S. COOPER

**2299. Interferometer for the measurement of sedimentation in a centrifuge.** J. W. Beams, N. Snidrow and H. M. Dixon, III (*Rev. Sci. Instrum.*, 1954, **25** [3], 295-296).—A modified Jamin interferometer method is described for the measurement of the refractive index in an ultracentrifuge cell that has a reliability comparable with the measurement of rotor speed and rotor temp.

G. SKIRROW

**2300. Microscope stage and integrating point counter for micrometric analysis of rocks.** I. H. Ford (*J. Sci. Instrum.*, 1954, **31** [5], 164-165).—A microscope stage is described which facilitates the accurate measurement of the areas occupied by component minerals in thin sections of rocks. The X-shift of the mechanical stage is fitted with a rotary switch, which automatically makes and breaks an electrical contact at regular intervals during the traverse of the stage. The pulses are recorded automatically on Post-Office type counters.

G. SKIRROW

## Thermal

**2301. Calorimeter thermometers.** British Standards Institution (B.S. 791:1954, 9 pp.).—Specifications are given for two series of mercury-in-glass thermometers: seven thermometers of 6° range, which together cover the range 9° to 33° C, and are suitable for use in bomb-calorimeters; and three thermometers of 12° range, together covering the range 8° to 32° C, suitable for use in other types of calorimeter. Thermometers required for gas calorimetry are not included. The thermometers are calibrated for total immersion. H. P. PAGET

**2302. A carbon-resistance furnace for the determination of gases in steel by the vacuum-fusion method.** R. M. Cook and G. E. Speight (*J. Iron & Steel Inst.*, 1954, **176** [3], 252-256).—The authors describe a vacuum-fusion apparatus that incorporates a carbon-resistance furnace instead of an H.F. furnace. The furnace has a split graphite heating element inside a water-cooled stainless-steel tube. Power is supplied from the mains through a small step-down transformer, thus obviating the need for an expensive high-frequency generator. The analytical system used provides for the rapid determination of oxygen and nitrogen *in situ*. Provision is also made for the determination of the extracted gases when required. The correct preparation of the CuO catalyst, which determines the accuracy of the method, is also given. The advantages of the apparatus are that it is easy to assemble, the temp. measurement can be obtained directly from the ammeter reading, Al- and Mn-steels do not have to be melted with tin and each determination takes only about  $\frac{1}{4}$  hr.

C. J. KEATTCH

## Electrical

**2303. Electrochemical methods of analysis.** J. Smelik (*Prakt. Chem.*, 1954, **5** [4], 86-88, 95).—The theoretical aspects of electrolytic analytical methods involving separation of metals are briefly outlined, and the principal uses and applications of such methods are described. Reference is made to their wide field of applicability—from micro-analysis and separation of traces of elements to separations of elements present in high concn.—and to various modifications of the method including electrolytic titration and the use of self-generated currents between two metals. M. TADMAN

**2304. Portable polarographic reference electrode.** R. K. Ladisch and S. L. Knesbach (*Anal. Chem.*, 1954, **26** [5], 941-942).—The construction of robust non-polarisable calomel reference electrodes suitable for routine polarography is described. The Hg is confined in a rigid support of fritted glass (max. pore-size 5 to 40  $\mu$ ), the area of Hg being equal to or several times that of the normal mercury pool. Calomel paste is placed in direct contact with the glass-frit so that contact between platinum wire (or W-Cu lead) is always undisturbed during polarographic procedure or frequent handling. Three types of such a cell are shown. The potential (after the electrodes have reached equilibrium overnight) is within 0.3 mV of the theoretical value, and the cells have been used for three months with excellent results. W. J. BAKER

**2305. Removal of dissolved oxygen by sodium sulphite during polarographic measurements.** S. Kikuchi, K. Honda and S. Kim (*Bull. Chem. Soc. Japan*, 1954, **27** [1], 65-68).—The effectiveness of  $\text{Na}_2\text{SO}_3$  in reducing dissolved O during polarographic determinations in phosphate, glycine-NaOH and borate-NaOH buffers, in 0.1 N LiCl, KCl and NaOH, and mixtures thereof with methanol was studied.  $\text{Na}_2\text{SO}_3$  exerts a max. effect in neutral solutions, becoming less effective at pH > 8; reduction occurs even in acid solutions. In alcohol-free solutions, the min. concn. of  $\text{Na}_2\text{SO}_3$  needed to reduce the original height of the O-wave by  $\approx$  98 per cent. within 5 min. vary from 0.02 per cent. (LiCl) to 0.3 per cent. (NaOH). In presence of 5 to 10 per cent. of an alcohol the effectiveness of  $\text{Na}_2\text{SO}_3$  is diminished unless  $\approx$  1 per cent. of  $\text{NaNO}_2$  is also present. Owing to the changing pH causing a shift in half-wave potential of org. compounds, calibration is necessary, even in buffered solutions, if the  $\text{Na}_2\text{SO}_3$  added is > 0.5 per cent. The freshly prepared solution of  $\text{Na}_2\text{SO}_3$  should not be used for about 10 min. after its preparation. W. J. BAKER

**2306. The use of the molybdenum electrode as indicator for hydrogen ions.** I. M. Issa and H. Khalifa (*Anal. Chim. Acta*, 1954, **10** [6], 567-574).—Polished Mo electrodes in buffer solutions at pH 2 to 12 show a max. drift amounting to 15 mV in 3 hr. When readings are taken 90 min. after immersion, there is an almost linear relation between pH and potential in the range pH 2 to 11, although appreciable deviation is noticed when the soln. is not aerated. The ratio of pH to mV is smaller than that of the hydrogen electrode and is affected by the constitution of the soln. and by the presence or absence of O. Both polished and oxidised electrodes yield sharply inflected curves in the titration of acids, but some appreciable end-point

errors are recorded. The  $\text{pK}_a$  values calculated from these titrations show, with some exceptions, fair agreement with published values.

W. C. JOHNSON

**2307. A simple electrical control for automatic Toepler pumps.** L. F. Hohnstedt and M. J. Steindler (*Rev. Sci. Instrum.*, 1954, **25** [3], 296-297).—Constructional details are given of an electrically operated automatic Toepler pump.

G. SKIRROW

**2308. A two-crystal gamma-ray scintillation spectrometer.** D. H. Peirson (*Nature*, 1954, **173**, 990-991).—The gamma-ray source is exposed simultaneously to NaI and anthracene scintillation counters. Compton continua from the two crystals are equalised in height and extent, and then subtracted in suitable counting-rate circuits so that each gamma-ray energy is represented only by a peak in the recorded spectrum. G. SKIRROW

**2309. Precision alpha-particle counting.** K. M. Glover and G. R. Hall (*Nature*, 1954, **173**, 991-992).—Results obtained in methods of absolute alpha-particle counting at Harwell and at the Radiation Laboratory of the University of California are compared. In spite of the difference in detail of the two methods, the agreement is good. G. SKIRROW

**2310. Measurement of the radioactivity of substances separated by chromatography and by electrophoresis.** P. Lerch and S. Neukomm (*Schweiz. med. Wochschr.*, 1954, **84** [18], 515-518).—An apparatus is described for the graphic registration of the radioactivity of isotope-marked materials separated by paper chromatography or paper electrophoresis. The apparatus is designed to give reproducible results with maximum sensitivity, the paper being fed slowly past a specially mounted Geiger-Müller tube, the output of which is integrated and fed to an ink-writing galvanometer. Details are given of the precision, probable error and sensitivity.

The application of this apparatus to the electrophoretically separated serum proteins marked with  $^{131}\text{I}$  indicate that (i) all the albumin is marked, (ii) only one form of  $^{131}\text{I}$ -albumin exists or can be separated, and (iii) the amount of hydrolysis is small. It has also been applied to the chromatogram of di-iodofluorescein marked with  $^{131}\text{I}$  run with acetone-water-conc.  $\text{NH}_3$  (80:18:2). Good agreement was attained between the autoradiogram and the integrator curve. On developing the chromatogram with starch, it was shown that not all the coloured zones were radioactive and not all the radioactive zones were coloured. Yellow areas were found to be due to non-iodinated fluorescein, and blue areas to  $\text{K}^{131}\text{I}$ . Chromatograms of  $\text{K}^{131}\text{I}$  indicated that the amount of KI used influenced the  $R_F$  value. This was approx. constant if up to  $10^{-6}$  g of material was used, but diminished (as much as 20 per cent.) when the amount was raised from  $10^{-6}$  to  $10^{-4}$  g. Furthermore, the radioactivity of the band was found distributed over a fairly narrow region and had a Gaussian distribution when small amounts were used, with a recovery of greater than 95 per cent. of the original material, but if the amount was large the distribution was over a wider area, the curve was flatter and showed two distinct peaks even with very pure KI. G. W. CAMBRIDGE

2311. Use of dissolved acetylene in liquid scintillation counters for the measurement of carbon-14 of low specific activity. B. N. Audric and J. V. P. Long (*Nature*, 1954, **173**, 992-993).—The effect of  $^{14}\text{C}$ -containing acetylene dissolved in toluene on the scintillation properties of a system at  $-78^\circ\text{C}$  has been investigated. The phosphor used was a soln. containing 3 g of 2:5-diphenyloxazole per litre of toluene containing 2 per cent. v/v of ethanol.

G. SKIRROW

2312. A fast neutron spectrometer. J. R. Holt and A. E. Litherland (*Rev. Sci. Instrum.*, 1954, **25** [3], 298).—Performance details are given for a spectrometer for neutrons of energy range 5 to 25 MeV.

G. SKIRROW

2313. Electromagnetic flowmeter. M. Robin (*J. Rech. Cent. Nat. Rech. Sci.*, 1953, **5**, 187-189).—A constant magnetic field, of intensity  $H$ , induces in a conducting liquid (Hg) of magnetic permeability  $\mu$ , flowing in a tube of diameter  $D$ , with a velocity  $V_m$  normal to the field, a max. potential drop given by  $10^{-8} \mu H D V_m$ . In the construction of a flowmeter based on this principle, the body of the meter is made of a cylinder of Plexiglas of external diameter 20 mm pierced with a channel of diameter 4 mm, joined to a stainless steel conduit of 4 mm internal diameter; the magnetic field is provided by an 8-mm air-gap between two permanent magnets made of an alloy (Ticonal) of high coercivity. The potential difference is measured by means of a potentiometer, provided with brass electrodes arranged directly opposite the Plexiglas body. Experimental results confirm the theory of the instrument.

PHYS. ABSTR.

ERRATA.—January (1954) issue, abstract 27, line 5.

For Y. J. Webber read T. J. Webber.

March (1954) issue, abstract 628, line 3.

For Waligóta read Waligóra.

## ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	micro-litre	$\mu$ l
ampere	amp.	micron	$\mu$
Angstrom unit	Å	milliampere	mA
anhydrous	anhyd.	milligram	mg
approximate, -ly	approx.	millilitre	ml
aqueous	aq.	millimetre	mm
atmospher-e, -ic	atm.	millivolt	mV
atomic	at.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	M
calculated	(calc.)	molecul -e, -ar	mol.
calorie (large)	kg-cal.	normal (concentration)	N
calorie (small)	g-cal.	number	no.
centimetre	cm	observed	(obs.)
coefficient	coeff.	organic	org.
concentrated	conc.	ounce	oz
concentration	concn.	part	pt.
constant	const.	patent	pat.
corrected	(corr.)	parts per million	p.p.m.
critical	crit.	per cent. wt. in wt.	per cent. w/w
crystalline	} cryst.	per cent. wt. in vol.	per cent. w/v
crystallised		per cent. vol. in vol.	per cent. v/v
cubic	cu.	potential difference	p.d.
current density	c.d.	pound	lb
cycles per second	c.p.s.	precipitate	ppt.
decompos -ing, -ition	(decomp.)	precipitated	pptd.
density	$\rho$	precipitating	pptg.
density, relative	d or wt. per ml	precipitation	pptn.
derivative	deriv.	preparation	prep.
dilute	dil.	qualitative, -ly	qual.
direct current	d.c.	quantitative, -ly	quant.
distilled	dist.	recrystallised	recryst.
electromotive force	e.m.f.	refractive index	$n_D^{25}$
electron-volt	eV	relative humidity	R.H.
equivalent	equiv.	revolutions per minute	r.p.m.
experiment, -al	expt.	saponification value	sap. val.
gram	g	saturated calomel electrode	S.C.E.
gram-molecule	mole	second (time)	sec.
half-wave potential	$E_p$	soluble	sol.
horse-power	h.p.	solution	soln.
hour	hr.	specific gravity	sp. gr.
hydrogen ion concentration	[H <sup>+</sup> ]	specific rotation	$[\alpha]_D^{25}$
hydrogen ion exponent	pH	square centimetre	sq. cm
inch	in.	standard temperature and pressure	s.t.p.
indefinite	indef.	temperature	temp.
infra-red	i.r.	ultra-violet	u.v.
insoluble	insol.	vapour density	v.d.
kilogram	kg	vapour pressure	v.p.
kilovolt	kV	volt	V
kilowatt	kW	volume	vol.
liquid	liq.	watt	W
maxim -um, -a	max.	wavelength	$\lambda$
melting-point	m.p.	weight	wt.
microgram	$\mu$ g		

In addition the following symbols are used—

greater than	>	less than	<
not greater than	≥	not less than	≤
is proportional to	∝	of the order of, approximately	≈

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, e.g., Cu<sup>+</sup>, Al<sup>+++</sup>, Cl<sup>-</sup>, SO<sub>4</sub><sup>++</sup>. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, e.g., ferric iron becomes Fe<sup>III</sup> and cuprous copper Cu<sup>I</sup>.

## ANALYTICAL ABSTRACTS

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